Supporting Information

Palladium-catalyzed amination oxidation of electron-rich olefins in green media

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Ⅰ. General Information

^{1}H, ^{13}C and ^{19}F NMR spectra were recorded at 400 MHz NMR spectrometer using $CDCl_3$ as solvent and TMS as an internal standard. Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). Coupling constants were reported in Hertz (Hz). IR spectra were obtained with an infrared spectrometer on either potassium bromide pellets or liquid films between two potassium bromide pellets. GC-MS data were obtained using electron ionization. HRMS was carried out on a high-resolution mass spectrometer (LCMS-IT-TOF). Melting points were measured using a melting point instrument and were uncorrected. TLC was performed using commercially available 100−400 mesh silica gel plates (GF_{254}). X-ray structural analyses were conducted on an X-ray analysis instrument. All the reaction temperatures reported are oil bath temperatures. Unless otherwise stated, all reagents and solvents were purchased from commercial suppliers and used without further purification.

Ⅱ. Experimental Procedure

I. General Procedure for the Synthesis of Substrates 2

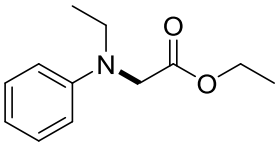
o-Alkynyltrifluoroacetanilides **1** were prepared according to the reported procedure.[1]

II. Typical Procedure for amination oxidation of olefin

To a 25 mL dried tube was added $Pd(OAc)_2$ (5 mol%), 5,5'-dimethyl-2,2'-bipyridine (6 mol%) and $[Bmim]BF_4$ (0.5 mL), and the mixture was stirred at room temperature for 0.5 h. Followed by the addition of aniline **1** (0.5 mmol), electron-rich alkenes **2** (1.0 mmol) and 35% aq. H_2O_2 (2 equiv). The mixture was stirred at room temperature for 12 h under an air atmosphere. After the reaction was completed, the mixture was extracted with EtOAc (10 mL x 3). The organic extract was washed with H_2O and dried over anhydrous Na_2SO_4. After removal of the EtOAc in vacuum, the crude product was obtained and purified by column chromatography on silica gel with hexanes or petroleum ether/ethyl acetate (10:1 to 50:1) to give the desired products **3**.

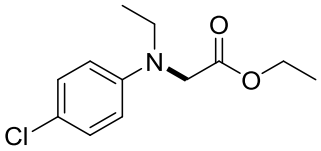
III. Characterization Data for All Products

Ethyl N-ethyl-N-phenylglycinate (3a)



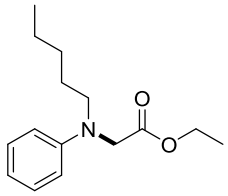
Yellow oil (94.2 mg, 91%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, J = 7.6 Hz, 2H), 6.70 (t, J = 7.2 Hz, 1H), 6.64 (d, J = 8.4 Hz, 2H), 4.18 (q, J = 7.2 Hz, 2H), 4.00 (s, 2H), 3.46 (q, J = 7.2 Hz, 2H), 1.25 (t, J = 7.0 Hz, 3H), 1.20 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 147.8, 129.2, 116.8, 112.0, 60.8, 52.3, 46.0, 14.2, 12.4 ppm. Vmax (KBr)/cm^{-1}: 3554, 3304, 3073, 2976, 1747f, 1605, 1508, 1192, 1027, 751. HRMS (ESI) m/z calculated for $[C_{11}H_{15}NO_2 + Na]^+$: 216.0995, found 216.0996.

Ethyl N-(4-chlorophenyl)-N-ethylglycinate (3b)



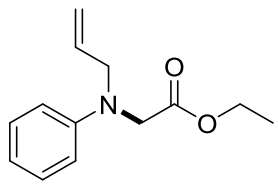
Yellow oil (86.8 mg, 72%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.15 (d, J = 9.1 Hz, 2H), 6.55 (d, J = 9.1 Hz, 2H), 4.19 (q, J = 7.2 Hz, 2H), 3.98 (s, 2H), 3.43 (q, J = 7.2 Hz, 2H), 1.26 (t, J = 7.0 Hz, 3H), 1.19 (t, J = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.9, 146.4, 128.9, 121.6, 113.1, 61.0, 52.3, 46.2, 14.1, 12.3 ppm. V_{max} (KBr)/cm^{-1}: 2971, 1741, 1501, 1192, 806. HRMS (ESI) m/z calculated for $[C_{12}H_{16}NO_2Cl + Na]^+$: 264.0762, found 264.0765.

Ethyl N-pentyl-N-phenylglycinateb (3c)



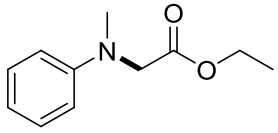
Yellow oil (76 mg, 61%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (t, J = 7.8 Hz, 2H), 6.71 (t, J = 7.2 Hz, 1H), 6.64 (d, J = 8.0 Hz, 2H), 4.20 (q, J = 7.2 Hz, 2H), 4.02 (s, 2H), 3.38 (t, J = 7.6 Hz, 2H), 1.65 (p, J = 7.4 Hz, 2H), 1.42-1.30 (m, 4H), 1.26 (t, J = 7.2 Hz, 3H), 0.93 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 148.1, 129.2, 116.8, 112.0, 60.8, 52.9, 52.1, 29.3, 27.2, 22.6, 14.2, 14.1 ppm. V_{max} (KBr)/cm^{-1}: 3054, 2941, 1742, 1600, 1501, 1370, 1186, 747. HRMS (ESI) m/z calculated for $[C_{15}H_{23}NO_2 + H]^+$: 250.1802, found 250.1807.

Ethyl N-allyl-N-phenylglycinate (3d)



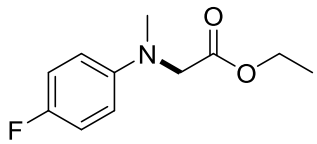
Yellow oil (46 mg, 42%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, J = 7.8 Hz, 2H), 6.73 (t, J = 7.2 Hz, 1H), 6.66 (d, J = 8.4 Hz, 2H), 5.89 (ddt, J = 15.7, 10.0, 5.0 Hz, 1H), 5.23 (d, J = 15.7 Hz, 1H), 5.17 (d, J = 10.0 Hz, 1H), 4.19 (q, J = 7.2 Hz, 2H), 4.02 (d, J = 5.0 Hz, 2H), 4.01 (s, 2H), 1.25 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 148.2, 133.9, 129.1, 117.3, 116.4, 112.4, 60.9, 54.2, 52.1, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 3596, 3460, 3366, 1737, 1505, 1179, 738. HRMS (ESI) m/z calculated for $[C_{13}H_{17}NO_2 + H]^+$: 220.1332, found 220.1341.

Ethyl N-methyl-N-phenylglycinate (3e)



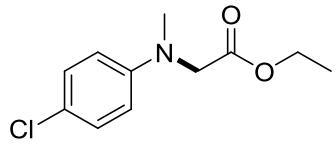
Yellow oil (75.3 mg, 78%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (t, J = 7.6 Hz, 2H), 6.73 (t, J = 7.2 Hz, 1H), 6.68 (d, J = 8.4 Hz, 2H), 4.15 (q, J = 7.2 Hz, 2H), 4.03 (s, 2H), 3.04 (s, 3H), 1.22 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 148.8, 129.1, 117.2, 112.2, 60.7, 54.4, 39.4, 14.1 ppm. V_{max} (KBr)/cm^{-1}: 2918, 1739, 1604, 1501, 1196, 751. HRMS (ESI) m/z calculated for $[C_{11}H_{15}NO_2 + Na]^+$: 216.0995, found 216.0996.

Ethyl N-ethyl-N-(4-fluorophenyl)glycinate (3f)



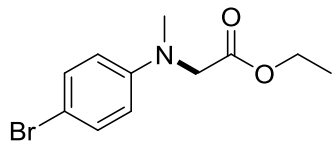
Yellow oil (63.3 mg, 60%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.93 (t, J = 8.4 Hz, 2H), 6.63 (dd, J = 8.6, 4.1 Hz, 2H), 4.17 (q, J = 7.2 Hz, 2H), 4.02 (s, 2H), 3.03 (s, 3H), 1.24 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 155.8 (d, J = 235.8 Hz), 145.5, 115.5 (d, J = 22.2 Hz), 113.5 (d, J = 7.4 Hz), 60.8, 55.0, 39. 9, 14.2. ^{19}F NMR (375 MHz, $CDCl_3$) δ -128.42 ppm. V_{max} (KBr)/cm^{-1}: 2973, 1842, 1739, 1514, 1208, 817. HRMS (ESI) m/z calculated for $[C_{11}H_{14}NO_2F + Na]^+$: 234.0901, found 234.0906.

Ethyl N-(4-chlorophenyl)-N-methylglycinate (3g)



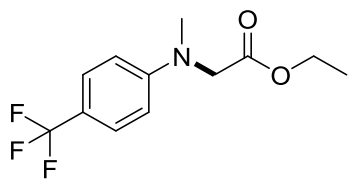
Yellow oil (62.4 mg, 55%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, J = 8.9 Hz, 2H), 6.59 (d, J = 8.9 Hz, 2H), 4.17 (q, J = 7.2 Hz, 2H), 4.02 (s, 2H), 3.03 (s, 3H), 1.24 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 147.5, 128.9, 122.1, 113.4, 60.9, 54.4, 39.5, 14.1 ppm. V_{max} (KBr)/cm^{-1}: 2974, 1739, 1500, 1203, 810. HRMS (ESI) m/z calculated for $[C_{11}H_{14}NO_2Cl + Na]^+$: 250.0605, found 250.0610.

ethyl N-(4-bromophenyl)-N-methylglycinate (3h)



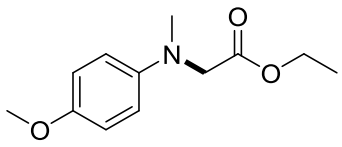
Yellow oil (67.8 mg, 50%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, J = 8.1 Hz, 2H), 6.54 (d, J = 8.1 Hz, 2H), 4.17 (q, J = 7.0 Hz, 2H), 4.02 (s, 2H), 3.03 (s, 3H), 1.24 (t, J = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 147.9, 131.8, 113.9, 109.3, 61.0, 54.4, 39.6, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 3366, 2950, 1738, 1493, 1199, 805. HRMS (ESI) m/z calculated for $[C_{11}H_{14}NO_2Br + Na]^+$: 294.0100, found 294.0104.

Ethyl N-methyl-N-(4-(trifluoromethyl)phenyl)glycinate (3i)



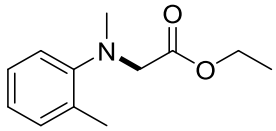
Yellow oil (54.8 mg, 42%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, J = 8.8Hz, 2H), 6.68 (d, J = 8.8 Hz, 2H), 4.19 (q, J = 7.2 Hz, 2H), 4.09 (s, 2H), 3.11 (s, 3H), 1.25 (t, J = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.2, 151.0, 126.5 (q, J = 3.8 Hz), 125.0 (d, J = 270.3 Hz), 119.1 - 118.2 (m), 111.3, 61.1, 54.1, 39.5, 29. 7, 14.1. ^{19}F NMR (475 MHz, $CDCl_3$) δ -61.04 ppm. V_{max} (KBr)/cm^{-1}: 3367, 2934, 1740, 1619, 1329, 1195, 1105, 817. HRMS (ESI) m/z calculated for $[C_{12}H_{14}NO_2F_3 + Na]^+$: 284.0869, found 284.0871.

Ethyl N-(4-methoxyphenyl)-N-methylglycinate (3j)



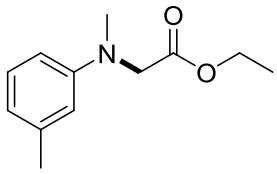
Yellow oil (53.5 mg, 48%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.82 (d, J = 9.0 Hz, 2H), 6.68 (d, J = 9.0 Hz, 2H), 4.16 (q, J = 7.2 Hz, 2H), 4.00 (s, 2H), 3.75 (s, 3H), 3.01 (s, 3H), 1.23 (t, J = 7.3 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.1, 152.0, 143.6, 114.7, 114.1, 60.7, 55.7, 55.3, 40.0, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 2958, 1740, 1508, 1196, 1031, 812. HRMS (ESI) m/z calculated for $[C_{12}H_{17}NO_3 + Na]^+$: 246.1101, found 246.1106.

Ethyl N-methyl-N-(o-tolyl)glycinate (3k)



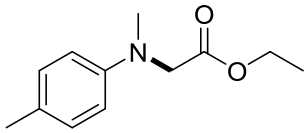
Yellow oil (83.8 mg, 81%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, J = 7.5 Hz, 2H), 7.11 (d, J = 7.3 Hz, 1H), 6.96 (t, J = 7.2 Hz, 1H), 4.17 (q, J = 7.1 Hz, 2H), 3.73 (s, 2H), 2.87 (s, 3H), 2.32 (s, 3H), 1.25 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 150.7, 132.0, 131.2, 126.3, 123.0, 120.2, 60.5, 57.4, 41.4, 18.3, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 2964, 1742, 1478, 1190, 750. HRMS (ESI) m/z calculated for $[C_{12}H_{17}NO_2 + Na]^+$: 230.1151, found 230.1156.

Ethyl N-methyl-N-(m-tolyl)glycinate (3l)



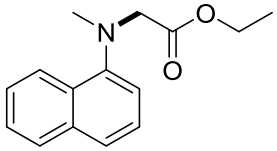
Yellow oil (64.2 mg, 62%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (t, J = 7.6 Hz, 1H), 6.60 (d, J = 7.6 Hz, 1H), 6.54 (s, 1H), 6.53 (d, J = 7.6 Hz, 1H), 4.20 (q, J = 7.1 Hz, 2H), 4.06 (s, 2H), 3.08 (s, 3H), 2.34 (s, 3H), 1.27 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 148.9, 138.7, 128.9, 118.2, 113.1, 109.5, 60.7, 54.5, 39.4, 21.8, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 2930, 1740, 1600, 1492, 1193,1029. HRMS (ESI) m/z calculated for $[C_{12}H_{17}NO_2 + Na]^+$: 230.1151, found 230.1158.

Ethyl N-methyl-N-(p-tolyl)glycinate (3m)



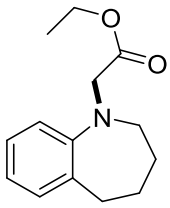
Yellow oil (76.6 mg, 74%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 (d, J = 8.0 Hz, 2H), 6.63 (d, J = 8.0 Hz, 2H), 4.17 (q, J = 7.2 Hz, 2H), 4.03 (s, 2H), 3.04 (s, 3H), 2.25 (s, 3H), 1.25 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 146.8, 129.7, 126.5, 112.6, 60.7, 54.7, 39.6, 20.2, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 2926, 1739, 1517, 1196, 806. HRMS (ESI) m/z calculated for $[C_{12}H_{17}NO_2 + Na]^+$: 230.1151, found 230.1158.

Ethyl N-methyl-N-(naphthalen-1-yl)glycinate (3n)



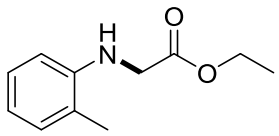
Yellow oil (48.6 mg, 40%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.23 (d, J = 8.2 Hz, 1H), 7.82 (dd, J = 7.5, 1.5 Hz, 1H), 7.55 (d, J = 8.2 Hz, 1H), 7.48 (dp, J = 6.8, 1.5 Hz, 2H), 7.39 (t, J = 7.8 Hz, 1H), 7.19 (d, J = 7.4 Hz, 1H), 4.21 (q, J = 7.2 Hz, 2H), 3.93 (s, 2H), 3.04 (s, 3H), 1.27 (t, J = 7.2 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.9, 148.7, 134.8, 128.7, 128.4, 125.8, 125.5, 125.5, 123.7, 123.5, 116.0, 60.6, 58.7, 41.8, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 2964, 1738, 1186, 755. HRMS (ESI) m/z calculated for $[C_{15}H_{17}NO_2 + N]^+$: 266.1151, found 266.1157.

Ethyl 2-(2,3,4,5-tetrahydro-1H-benzo[b]azepin-1-yl)acetate (3o)



Yellow oil (58.3 mg, 50%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14-7.07 (m, 2H), 6.86 (dt, J = 7.4, 1.2 Hz, 1H), 6.78 (dd, J = 8.4, 1.2 Hz, 1H), 4.23 (q, J = 7.1 Hz, 2H), 3.98 (s, 2H), 3.24 - 3.03 (m, 2H), 2.90 - 2.68 (m, 2H), 1.88-1.72 (m, 2H), 1.72-1.60 (m, 2H), 1.30 (t, J = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 151.0, 135.3, 130.3, 126.6, 121.3, 117.3, 60.7, 56.4, 54.7, 35.0, 29.5, 25.6, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 2926, 1739, 1491, 1177, 750. HRMS (ESI) m/z calculated for $[C_{14}H_{19}NO_2 + H]^+$: 234.1489, found 234.1491.

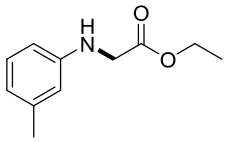
Ethyl o-tolylglycinate (3p)



Yellow oil (54 mg, 56%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, J = 7.7 Hz, 1H), 7.10 (d, J = 7.3 Hz, 1H), 6.73 (t, J = 7.4 Hz, 1H), 6.51 (d, J = 8.0 Hz, 1H), 4.28 (q, J = 7.1 Hz, 2H), 3.96 (s, 2H), 2.24 (s, 3H), 1.33 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 145.0, 130.2, 127.1, 122.4, 117.7, 109.8, 61.3, 45.8, 17.3, 14.1 ppm. V_{max} (KBr)/cm^{-1}: 3417, 2925, 1733, 1513, 1193, 1021, 747. HRMS (ESI) m/z calculated for $[C_{11}H_{15}NO_2 + Na]^+$: 216.0995, found 216.0993.

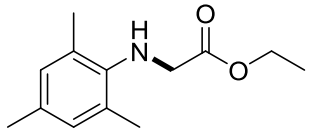
Ethyl m-tolylglycinate (3q)

Yellow oil (51.2 mg, 53%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09 (t, J = 7.6 Hz, 1H), 6.59 (d, J = 7.4



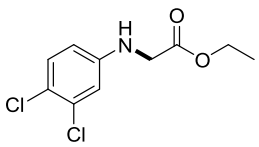
Hz, 1H), 6.43 (d, J = 9.9 Hz, 2H), 4.25 (q, J = 7.0 Hz, 2H), 3.90 (s, 3H), 2.29 (s, 3H), 1.30 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 147.1, 139.1, 129.1, 119.1, 113.8, 110.1, 61.2, 45.9, 21.6, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 3370, 2926, 1734, 1524, 761. HRMS (ESI) m/z calculated for $[C_{11}H_{15}NO_2 + Na]^+$: 216.0995, found 216.0994.

Ethyl mesitylglycinate (3r)



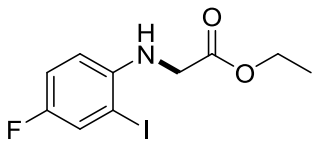
Yellow oil (76.3 mg, 69%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.85 (s, 2H), 4.25 (q, J = 7.1 Hz, 2H), 3.80 (s, 1H), 3.68 (s, 1H), 2.34 (s, 6H), 2.27 (s, 3H), 1.32 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 143.0, 131.1, 129.4, 128.8, 61.0, 50.1, 20.4, 18.4, 14.1 ppm. V_{max} (KBr)/cm^{-1}: 2927, 1665, 1527, 755. HRMS (ESI) m/z calculated for $[C_{13}H_{19}NO_2 + H]^+$: 222.1489, found 222.1491.

Ethyl (3,4-dichlorophenyl)glycinate (3s)



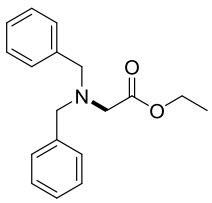
Yellow oil (55.6 mg, 45%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, J = 8.7 Hz, 1H), 6.65 (d, J = 2.6 Hz, 1H), 6.44 (dd, J = 8.7, 2.6 Hz, 1H), 4.39 (br, 1H), 4.25 (q, J = 7.1 Hz, 2H), 3.85 (s, 2H), 1.30 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 146.9, 133.4, 131.2, 121.2, 114.5, 113.2, 62.1, 46.0, 14.6 ppm. V_{max} (KBr)/cm^{-1}: 3377, 1715, 1473, 1244. HRMS (ESI) m/z calculated for [$C_{10}H_{11}NO_2Cl_2$ + Na]$^{+}$: 270.0059, found 270.0052.

Ethyl (4-fluoro-2-iodophenyl)glycinate (3t).



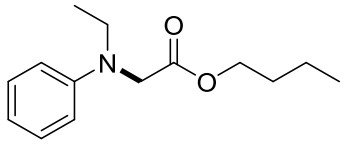
Yellow oil (64.4 mg, 40%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (dd, J = 7.8, 2.7 Hz, 1H), 6.96 (td, J = 8.6, 2.6 Hz, 1H), 6.36 (dd, J = 8.9, 4.7 Hz, 1H), 4.67 (s, 1H), 4.26 (q, J = 7.1 Hz, 2H), 3.90 (d, J = 5.2 Hz, 2H), 1.30 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 156.3, 153.9, 143.2, 125.8 (d, J = 24.0 Hz), 115.9 (d, J = 22.0 Hz), 110.4 (d, J = 8.0 Hz), 83.9 (d, J = 9.0 Hz), 61.5, 46.6, 14.2 ppm. ^{19}F NMR (471 MHz, $CDCl_3$) δ -126.2. V_{max} (KBr)/cm^{-1}: 2843, 1724, 1466, 1190, 723. HRMS (ESI) m/z calculated for [$C_{10}H_{11}NO_2FI$ + Na]$^{+}$: 345.9711, found 345.9715.

Ethyl dibenzylglycinate (3u)



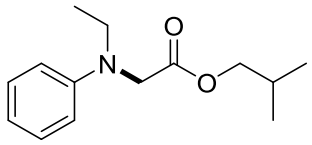
Yellow oil (101.9 mg, 72%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, J =7.5 Hz, 4H), 7.30 (t, J = 7.6 Hz, 4H), 7.22 (t, J = 7.3 Hz, 2H), 4.13 (q, J = 7.2 Hz, 2H), 3.80 (s, 4H), 3.27 (s, 2H), 1.24 (t, J = 7.2 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.3, 139.0, 128.8, 128.2, 127.0, 60.1, 57.7, 53.5, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 2843, 1724, 1466, 1190, 723. HRMS (ESI) m/z calculated for [$C_{18}H_{21}NO_2$ + H]$^{+}$: 284.1645, found 284.1650.

Butyl N-ethyl-N-phenylglycinate (3w)



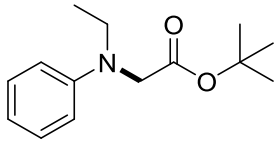
Yellow oil (99.9 mg, 85%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, J = 7.4 Hz, 2H), 6.70 (t, J = 7.3 Hz, 1H), 6.64 (d, J = 7.9 Hz, 2H), 4.13 (t, J = 6.6 Hz, 2H), 4.01 (s, 2H), 3.46 (q, J = 7.0 Hz, 2H), 1.59 (p, J = 7.0 Hz, 2H), 1.33 (h, J = 7.4 Hz, 2H), 1.20 (t, J = 7.0 Hz, 3H), 0.90 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 147.7, 129.2, 116.8, 112.0, 64.7, 52.3, 46.1, 30.5, 19.0, 13.6, 12.4 ppm. V_{max} (KBr)/cm^{-1}: 2954, 1741, 1599, 1502, 1367, 1188, 749. HRMS (ESI) m/z calculated for $[C_{14}H_{21}NO_2 + Na]^+$: 258.1464, found 258.1471.

Isobutyl N-ethyl-N-phenylglycinate (3x)



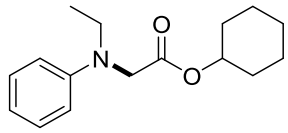
Yellow oil (89.3mg, 76%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, J = 7.4 Hz, 2H), 6.70 (t, J = 7.2 Hz, 1H), 6.65 (d, J = 7.9 Hz, 2H), 4.02 (s, 2H), 3.91 (d, J = 6.5 Hz, 2H), 3.46 (q, J = 7.0 Hz, 2H), 1.90 (dt, J = 13.2, 6.6 Hz, 1H), 1.21 (t, J = 7.0 Hz, 3H), 0.88 (d, J = 6.6 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 147.8, 129.2, 116.8, 112.0, 70.9, 52.3, 46.1, 27.7, 18.9, 12.5 ppm. V_{max} (KBr)/cm^{-1}: 2955, 1740, 1187, 746. HRMS (ESI) m/z calculated for $[C_{14}H_{21}NO_2 + Na]^+$: 258.1464, found 258.1458.

Tert-butyl N-ethyl-N-phenylglycinate (3y)



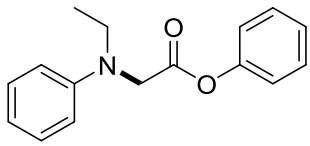
Yellow oil (52.9 mg, 45%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (t, J = 7.3 Hz, 2H), 6.70 (t, J = 7.2 Hz, 1H), 6.65 (d, J = 7.9 Hz, 2H), 3.91 (s, 2H), 3.47 (q, J = 7.0 Hz, 2H), 1.45 (s, 9H), 1.22 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 147.9, 129.1, 116.6, 111.9, 81.3, 53.2, 46.1, 28.0, 12.5 ppm. V_{max} (KBr)/cm^{-1}: 2961, 1734, 1600, 1500, 1367, 1154, 745. HRMS (ESI) m/z calculated for $[C_{14}H_{21}NO_2 + Na]^+$: 258.1464, found 258.1468.

Cyclohexyl N-ethyl-N-phenylglycinate (3z)



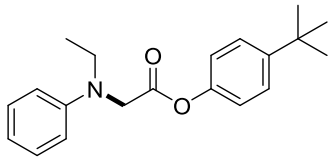
Yellow oil (95.3 mg, 73%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22 (dd, J = 8.8, 7.3 Hz, 2H), 6.72 (t, J = 7.3 Hz, 1H), 6.66 (d, J = 8.0 Hz, 2H), 4.85 (ddt, J = 8.8, 4.8, 4.3 Hz, 1H), 4.01 (s, 2H), 3.48 (q, J = 7.1 Hz, 2H), 1.88 - 1.76 (m, 2H), 1.73 - 1.60 (m, 2H), 1.54 - 1.28 (m, 6H), 1.23 (t, J = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.8, 147.8, 129.1, 116.7, 112.0, 73.2, 52.7, 46.1, 31.4, 25.3, 23.4, 12.5 ppm. V_{max} (KBr)/cm^{-1}: 2937, 1736, 1601, 1500, 1364, 1195, 871, 747. HRMS (ESI) m/z calculated for $[C_{16}H_{23}NO_2 + Na]^+$: 284.1621, found 284.1624.

Phenyl N-ethyl-N-phenylglycinate (3aa)



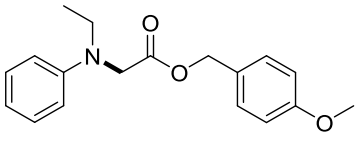
Yellow oil (66.3 mg, 52%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (t, J = 7.6 Hz, 2H), 7.25 (t, J = 7.9 Hz, 3H), 7.06 (d, J = 8.0 Hz, 2H), 6.83-6.68 (m, 3H), 4.26 (s, 2H), 3.55 (q, J = 7.1 Hz, 2H), 1.26 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 150.5, 147.6, 129.4, 129.4, 125.9, 121.3, 117.2, 112.2, 52.6, 46.2, 29.7, 12.6 ppm. V_{max} (KBr)/cm^{-1}: 3371, 2975, 1751, 1487, 1146, 738. HRMS (ESI) m/z calculated for $[C_{16}H_{17}NO_2 + Na]^+$: 278.1151, found 278.1150.

4-(tert-butyl)phenyl N-ethyl-N-phenylglycinate (3ab)



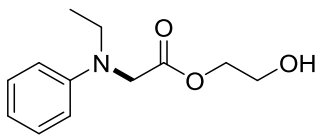
Yellow oil (77.8 mg, 50%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, J = 8.7 Hz, 2H), 7.26-7.20 (m, 2H), 6.98 (d, J = 8.7 Hz, 2H), 6.78-6.70 (m, 3H), 4.25 (s, 2H), 3.54 (q, J = 7.1 Hz, 2H), 1.29 (s, 9H), 1.26 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 148.8, 148.2, 147.7, 129.3, 126.3, 120.6, 117.2, 112.3, 52.6, 46.2, 34.4, 31.4, 12.6 ppm. V_{max} (KBr)/cm^{-1}: 2956, 1763, 1600, 1503. 1182, 744. HRMS (ESI) m/z calculated for $[C_{20}H_{25}NO_2 + H]^+$: 312.1958, found 312.1960.

4-methoxybenzyl N-ethyl-N-phenylglycinate (3ac)



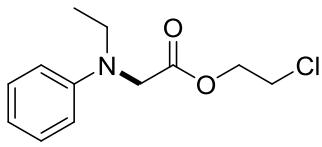
Yellow oil (109 mg, 73%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22 (d, J = 8.7 Hz, 2H), 7.19-7.14 (m, 2H), 6.88-6.81 (m, 2H), 6.69 (t, J = 7.3 Hz, 1H), 6.61 (d, J = 8.0 Hz, 2H), 4.01 (s, 2H), 3.76 (s, 3H), 3.43 (q, J = 7.1 Hz, 2H), 1.16 (t, J = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.2, 159.6, 147.6, 130.0, 129.1, 127.7, 116.8, 113.8, 112.0, 66.3, 55.1, 52.2, 46.0, 12.4 ppm. V_{max} (KBr)/cm^{-1}: 2946, 1740, 1604, 1489, 1152, 980, 831, 535. HRMS (ESI) m/z calculated for $[C_{18}H_{21}NO_3$+ Na$]^+$: 322.1414, found 322.1418.

2-hydroxyethyl N-ethyl-N-phenylglycinate (3ad)



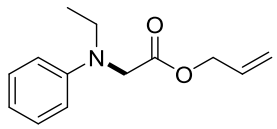
Yellow oil (79.2 mg, 71%); ^{1}H NMR (400 MHz, DMSO) δ 7.14 (t, J = 7.4 Hz, 2H), 6.60 (t, J = 8.4 Hz, 1H), 6.59 (d, J = 8.4 Hz, 2H), 4.83 (t, J = 5.5 Hz, 1H), 4.12 (s, 2H), 4.09 (t, J = 4.5 Hz, 2H), 3.58 (dd, J = 12.5, 6.0 Hz, 2H), 3.40 (q, J = 6.9 Hz, 2H), 1.11 (t, J = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, DMSO) δ 171.1, 147.7, 129.1, 116.0, 111.6, 66.1, 59.1, 51.4, 45.3, 12.4 ppm. V_{max} (KBr)/cm^{-1}: 2949, 1737, 1502, 1187, 748. HRMS (ESI) m/z calculated for $[C_{12}H_{17}NO_3$ + Na$]^+$: 246.1104, found 246.1105.

2-chloroethyl N-ethyl-N-phenylglycinate (3ae)



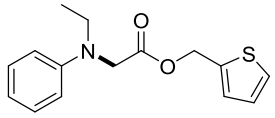
Yellow oil (94 mg, 78%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (t, J = 7.5 Hz, 2H), 6.71 (t, J = 7.2 Hz, 1H), 6.64 (d, J = 7.9 Hz, 2H), 4.35 (t, J = 5.6 Hz, 2H), 4.05 (s, 2H), 3.62 (t, J = 5.6 Hz, 2H), 3.45 (q, J = 7.0 Hz, 2H), 1.20 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 147.6, 129.2, 116.9, 112.0, 64.2, 52.0, 46.0, 41.4, 12.4 ppm. V_{max} (KBr)/cm^{-1}: 2960, 1747, 1600, 1501, 1181, 1010, 752. HRMS (ESI) m/z calculated for $[C_{12}H_{16}NO_2Cl$ + Na$]^+$: 264.0762, found 264.0760.

Allyl N-ethyl-N-phenylglycinate (3af)



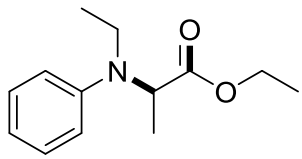
Yellow oil (61.4 mg, 56%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (t, J = 7.4 Hz, 2H), 6.71 (t, J = 7.3 Hz, 1H), 6.65 (d, J = 7.9 Hz, 2H), 5.90 (ddt, J = 16.7, 11.2, 5.7 Hz, 1H), 5.26 (dd, J = 26.1, 13.8 Hz, 2H), 4.63 (d, J = 5.6 Hz, 2H), 4.05 (s, 2H), 3.47 (q, J = 7.0 Hz, 2H), 1.21 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 147.7, 131.8, 129.2, 118.5, 116.9, 112.1, 65.5, 52.2, 46.0, 12.4 ppm. V_{max} (KBr)/cm^{-1}: 2948, 1743, 1600, 1501, 1368, 1179, 981, 747. HRMS (ESI) m/z calculated for $[C_{13}H_{17}NO_2 + Na]^+$: 242.1151, found 242.1156.

thiophen-2-ylmethyl N-ethyl-N-phenylglycinate (3ag)



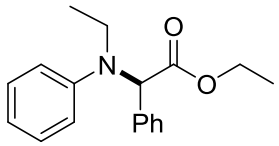
colorless oil (113 mg, 82%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (dd, J = 5.1, 1.1 Hz, 1H), 7.19 (dd, J = 8.8, 7.2 Hz, 2H), 7.06 (dd, J = 3.5, 1.1 Hz, 1H), 6.96 (dd, J = 5.1, 3.5 Hz, 1H), 6.70 (t, J = 7.3 Hz, 1H), 6.62 (d, J = 8.2 Hz, 2H), 5.31 (s, 2H), 4.03 (s, 2H), 3.44 (q, J = 7.1 Hz, 2H), 1.18 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 147.6, 137.5, 129.2, 128.3, 126.9, 126.8, 116.9, 112.1, 60.8, 52.2, 46.0, 12.4 ppm. HRMS (ESI) m/z calculated for $[C_{15}H_{17}NO_2S + H]^+$: 276.1048, found 276.1054.

Ethyl N-ethyl-N-phenylalaninate (3aj)



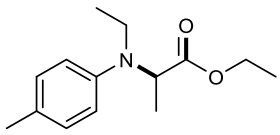
Yellow oil (48.6 mg, 44%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (t, J = 7.9 Hz, 2H), 6.85-6.70 (m, 3H), 4.48 (q, J = 7.2 Hz, 1H), 4.15 (qt, J = 7.3 ,3.7 Hz, 2H), 3.40 (q, J = 7.0 Hz, 2H), 1.51 (d, J = 7.2 Hz, 3H), 1.23 (t, J = 7.1 Hz, 3H), 1.20 (t, J = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.7, 148.1, 129.1, 117.2, 113.6, 60.7, 57.1 41.5, 15.9, 14.4, 14.1 ppm. V_{max} (KBr)/cm^{-1}: 2973, 1732, 1598, 1500, 1190, 750. HRMS (ESI) m/z calculated for $[C_{13}H_{19}NO_2 + H]^+$: 222.1489, found 222.1493.

Ethyl 2-(ethyl(phenyl)amino)-2-phenylacetate (3ak)



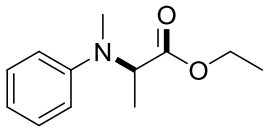
Yellow oil (32.5 mg, 23%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.32 (m, 5H), 7.25 (t, J = 8.0 Hz, 2H), 6.86 (d, J = 8.2 Hz, 2H), 6.79 (t, J = 7.3 Hz, 1H), 5.52 (s, 1H), 4.23 (qq, J = 10.8, 7.1 Hz, 2H), 3.33 (d, J = 7.0 Hz, 2H), 1.23 (t, J = 7.1 Hz, 3H), 0.91 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.0, 148.5, 136.1, 129.2, 128.9, 128.5, 128.1, 118.2, 114.5, 66.3, 61.1, 42.5, 14.2, 13.4 ppm. V_{max} (KBr)/cm^{-1}: 2955, 1735, 1592, 1493, 1173, 748. HRMS (ESI) m/z calculated for $[C_{18}H_{21}NO_2 + H]^+$: 284.1640, found 284.1645.

ethyl *N*-ethyl-*N*-(*p*-tolyl)alaninate (3al)



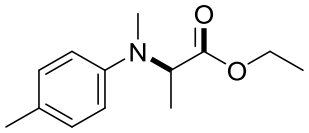
Yellow oil (56.4 mg, 77%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (d, J = 8.4 Hz, 2H), 6.69 (d, J = 8.4 Hz, 2H), 4.36 (q, J = 7.2 Hz, 1H), 4.16 (pt, J = 8.0, 4.4 Hz, 2H), 3.36 (q, J = 7.2 Hz, 2H), 2.25 (s, 3H), 1.48 (d, J = 7.2 Hz, 3H), 1.23 (t, J = 7.2 Hz, 3H), 1.17 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 146.0, 129.7, 126.8, 114.4, 60.8, 57.6, 41.7, 20.3, 15.9, 14.5, 14.2. HRMS (ESI) m/z calculated for $[C_{14}H_{21}NO_2 + H]^+$: 236.1645, found 236.1641.

ethyl N-methyl-N-phenylalaninate (3am)



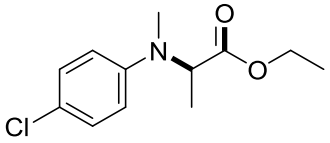
Yellow oil (79.7 mg, 77%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.09 (m, 2H), 6.89-6.62 (m, 3H), 4.50 (q, J = 7.1 Hz, 1H), 4.22-4.10 (m, 2H), 2.89 (s, 3H), 1.47 (d, J = 7.1 Hz, 3H), 1.22 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 149.6, 129.1, 117.6, 113.4, 60.8, 57.1, 33.0, 15.2, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 2981, 2820, 1734, 1599, 1502, 1375, 1196, 1118, 651, 691. HRMS (ESI) m/z calculated for $[C_{12}H_{17}NO_2 + H]^+$: 208.1331, found 208.1332.

ethyl *N*-methyl-*N*-(*p*-tolyl)alaninate (3an)



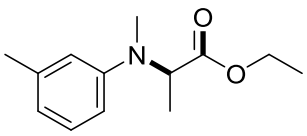
Yellow oil (51.9 mg, 47%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 (d, J = 8.4 Hz, 2H), 6.73 (d, J = 8.4 Hz, 2H), 4.45 (q, J = 7.2 Hz, 1H), 4.16 (dq, J = 7.2, 5.7 Hz, 2H), 2.87 (s, 3H), 2.25 (s, 3H), 1.45 (d, J = 7.2 Hz, 3H), 1.23 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5, 147.7, 129.7, 127.0, 113.9, 60.7, 57.5, 33.1, 20.3, 15.1, 14.3. HRMS (ESI) m/z calculated for $[C_{13}H_{19}NO_2 + H]^+$: 222.1489, found 222.1486.

ethyl *N*-(4-chlorophenyl)-*N*-methylalaninate (3ao)



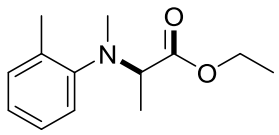
Yellow oil (42.2 mg, 35%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, J = 8.8 Hz, 2H), 6.70 (d, J = 8.8 Hz, 2H), 4.43 (q, J = 7.2 Hz, 1H), 4.16 (dq, J = 7.2, 4.0 Hz, 2H), 2.87 (s, 3H), 1.47 (d, J = 7.2 Hz, 3H), 1.23 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.0, 148.4, 128.9, 114.6, 60.9, 57.3, 33.1, 15.3, 14.3. HRMS (ESI) m/z calculated for $[C_{12}H_{18}ClNO_2 + H]^+$: 242.0942, found 242.0939.

ethyl *N*-methyl-*N*-(*m*-tolyl)alaninate (3ap)



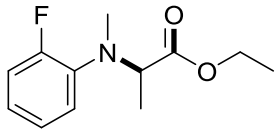
Yellow oil (55.1 mg, 50%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (dt, J = 7.2, 2.0 Hz, 2H), 6.70-6.56 (m, 3H), 4.53 (q, J = 7.2 Hz, 1H), 4.28-4.12 (m, 2H), 2.91 (s, 3H), 2.34 (s, 3H), 1.49 (d, J = 7.2 Hz, 3H), 1.27 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.4, 149.8, 138.8, 129.0, 118.5, 114.2, 110.6, 60.8, 57.1, 33.0, 21.9, 15.2, 14.3. HRMS (ESI) m/z calculated for $[C_{13}H_{19}NO_2 + H]^+$: 222.1489, found 222.1485.

ethyl N-methyl-N-(o-tolyl)alaninate (3aq)



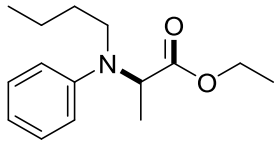
Yellow oil (51.9mg, 47%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21-7.08 (m, 3H), 6.97 (td, J = 7.1, 2.0 Hz, 1H), 4.20-4.05 (m, 2H), 3.82 (q, J = 7.1 Hz, 1H), 2.77 (s, 3H), 2.31 (s, 3H), 1.40 (d, J = 7.1 Hz, 3H), 1.23 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 151.0, 133.0, 131.1, 126.1, 123.1, 121.8, 60.4, 60.3, 35.3, 18.3, 14.7, 14.2 ppm. V_{max} (KBr)/cm^{-1}: 3450, 2937, 1732, 1476, 11373, 1266, 1100, 863, 754, 463. HRMS (ESI) m/z calculated for $[C_{13}H_{19}NO_2 + H]^+$: 222.1489, found 222.1485.

ethyl *N*-(2-fluorophenyl)-*N*-methylalaninate (3ar)



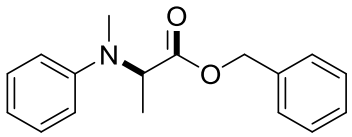
Yellow oil (65.2mg, 58%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.07-6.94 (m, 3H), 6.92-6.83 (m, 1H), 4.27 (qd, J = 7.2, 1.6 Hz, 1H), 4.13 (qq, J = 10.8, 7.2 Hz, 2H), 2.83 (s, 3H), 1.46 (d, J = 7.2 Hz, 3H), 1.21 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.0, 155.3 (d, J = 244 Hz), 139.5 (d, J = 8.3 Hz), 124.2 (d, J = 3.4 Hz), 121.7 (d, J = 7.9 Hz), 120.6 (d, J = 3.2 Hz), 116.1 (d, J = 21 Hz), 60.6, 59.7, 59.6, 33.9 (d, J = 1.5 Hz), 15.1, 14.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ -122.6 (ddd, J = 12.9, 8.1, 4.7 Hz, 1F). HRMS (ESI) m/z calculated for $[C_{12}H_{16}FNO_2 + H]^+$: 226.1238, found 226.1234.

ethyl *N*-butyl-*N*-phenylalaninate (3as)



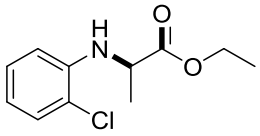
Yellow oil (87.2 mg, 70%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.19 (m, 2H), 6.79-6.71 (m, 3H), 4.41 (q, J = 7.1 Hz, 2H), 4.17 (dq, J = 7.2, 4.0 Hz, 2H), 3.29 (dd, J = 8.8, 6.8 Hz, 2H), 1.70-1.53 (m, 2H), 1.51 (d, J = 7.2 Hz, 3H), 1.37 (h, J = 7.2 Hz, 2H), 1.24 (d, J = 7.2 Hz, 3H), 0.96 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 148.5, 129.1, 117.4, 113.9, 60.8, 57.6, 47.4, 31.0, 20.3, 15.9, 14.2, 14.0. HRMS (ESI) m/z calculated for $[C_{15}H_{23}NO_2 + H]^+$: 250.1802, found 250.1797.

benzyl N-methyl-N-phenylalaninate (3au)



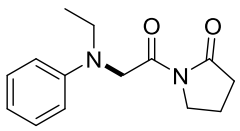
Yellow oil (101 mg, 75%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.14 (m, 7H), 6.80-6.74 (m, 3H), 5.16 (d, J =12.4 Hz, 1H), 5.11 (d, J =12.4 Hz, 1H), 4.57 (q, J = 7.1 Hz, 1H), 2.88 (s, 3H), 1.49 (d, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2, 149.7, 135.8, 129.1, 128.5, 128.2, 128.0, 66.5, 57.2, 33.0, 15.3 ppm. V_{max} (KBr)/cm^{-1}:3479, 3393, 2921, 1735, 1598, 1189, 750. HRMS (ESI) m/z calculated for $[C_{17}H_{19}NO_2 + H]^+$: 270.1488, found 270.1489.

ethyl (2-chlorophenyl)alaninate (3av)



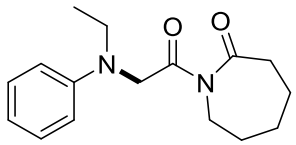
Yellow oil (68.3 mg, 60%);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.22 (m, 1H), 7.11 (ddd, J = 8.0, 7.3, 1.5 Hz, 1H), 6.66 (dt, J = 6.2, 1.4 Hz, 1H), 6.57 (dd, J = 8.1, 1.4 Hz, 1H), 4.20 (q, J = 7.1 Hz, 2H), 4.15 (q, J = 6.8 Hz, 1H), 1.53 (d, J = 6.9 Hz, 3H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 142.6, 129.4, 127.7, 119.7, 118.1, 111.6, 61.3, 51.8, 18.8, 14.1 ppm. V_{max} (KBr)/cm^{-1}: 3398, 2977, 1736, 1601, 1507, 1309, 1175, 1036, 744. HRMS (ESI) m/z calculated for $[C_{11}H_{14}NO_2Cl + H]^+$: 228.0784, found 228.0786.

1-(N-ethyl-N-phenylglycyl)pyrrolidin-2-one (5a)



Yellow oil (84 mg, 68%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (t, J = 7.4 Hz, 2H), 6.68 (t, J = 7.2 Hz, 1H), 6.61 (d, J = 7.9 Hz, 2H), 4.64 (s, 2H), 3.82 (t, J = 7.1 Hz, 2H), 3.44 (q, J = 7.0 Hz, 2H), 2.62 (t, J = 8.0 Hz, 2H), 2.08 (p, J = 7.5 Hz, 2H), 1.22 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.0, 171.6, 147.8, 129.0, 116.4, 111.8, 55.1, 45.8, 45.1, 33.2, 17.6, 12.5 ppm. V_{max} (KBr)/cm^{-1}: 2968, 1718, 1373, 1237, 756. HRMS (ESI) m/z calculated for $[C_{14}H_{18}N_2O_2 + Na]^+$: 269.1260, found 269.1264.

1-(N-ethyl-N-phenylglycyl)azepan-2-one (5b)

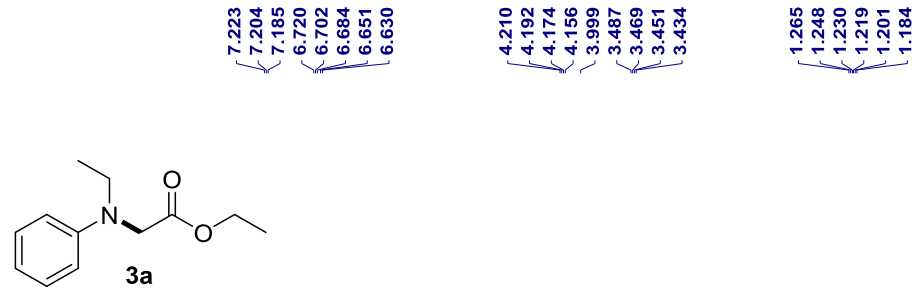


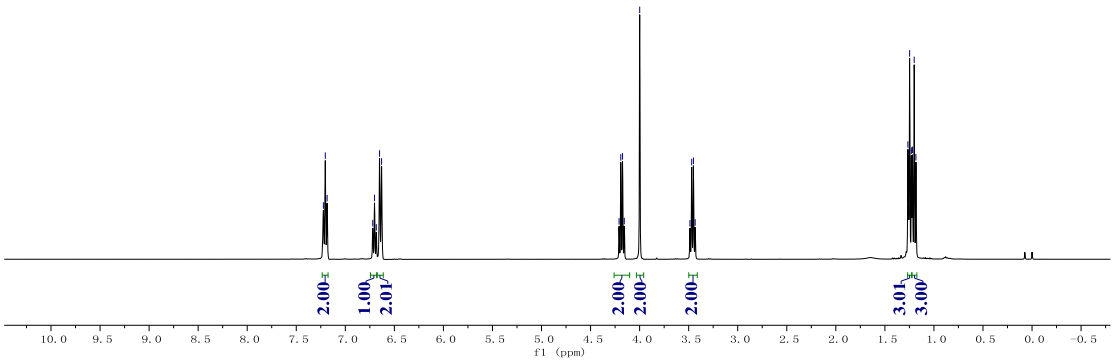
Yellow oil (92 mg, 67%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (t, J = 7.5 Hz, 2H), 6.67 (t, J = 7.2 Hz, 1H), 6.57 (d, J = 8.0 Hz, 2H), 4.56 (s, 2H), 3.90 (d, J = 8.2 Hz, 2H), 3.43 (q, J = 7.0 Hz, 2H), 2.80 - 2.71 (m, 2H), 1.88 - 1.65 (m, 6H), 1.20 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 174.1, 147.9, 129.1, 116.3, 111.7, 57.2, 46.0, 43.4, 39.4, 29.1, 28.6, 23.6, 12.6 ppm. V_{max} (KBr)/cm^{-1}: 2934, 1697, 1508, 1177, 974, 749. HRMS (ESI) m/z calculated for $[C_{16}H_{22}N_2O_2 + Na]^+$: 297.1573, found 297.1579.

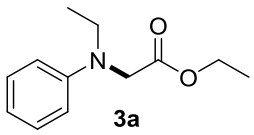
Reference

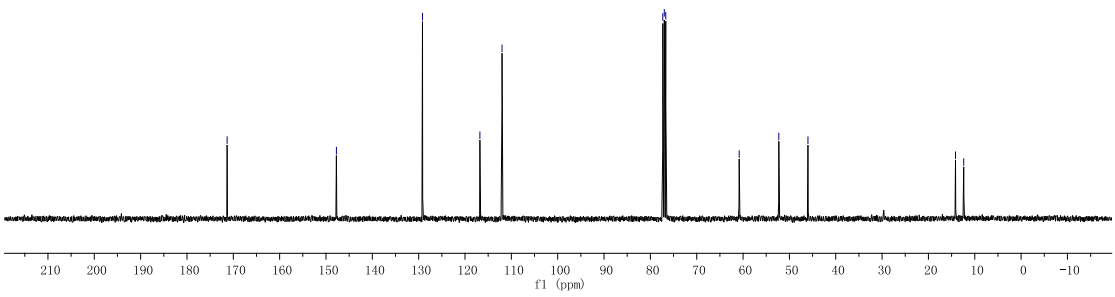
1. Cacchi, S.; Fabrizi, G.; Pace, P. *J. Org. Chem.* **1998**, *63*, 1001.
2. (a) Watson, D. J.; Dowdy, E. D.; Li, W.; Wang, J.; Polniaszek, R. *Tetrahedron Lett.* **2001**, *42*, 1827. (b) Schleicher, K. D.; Jamison, T. F. *Org. Lett.* **2007**, *9*, 87.

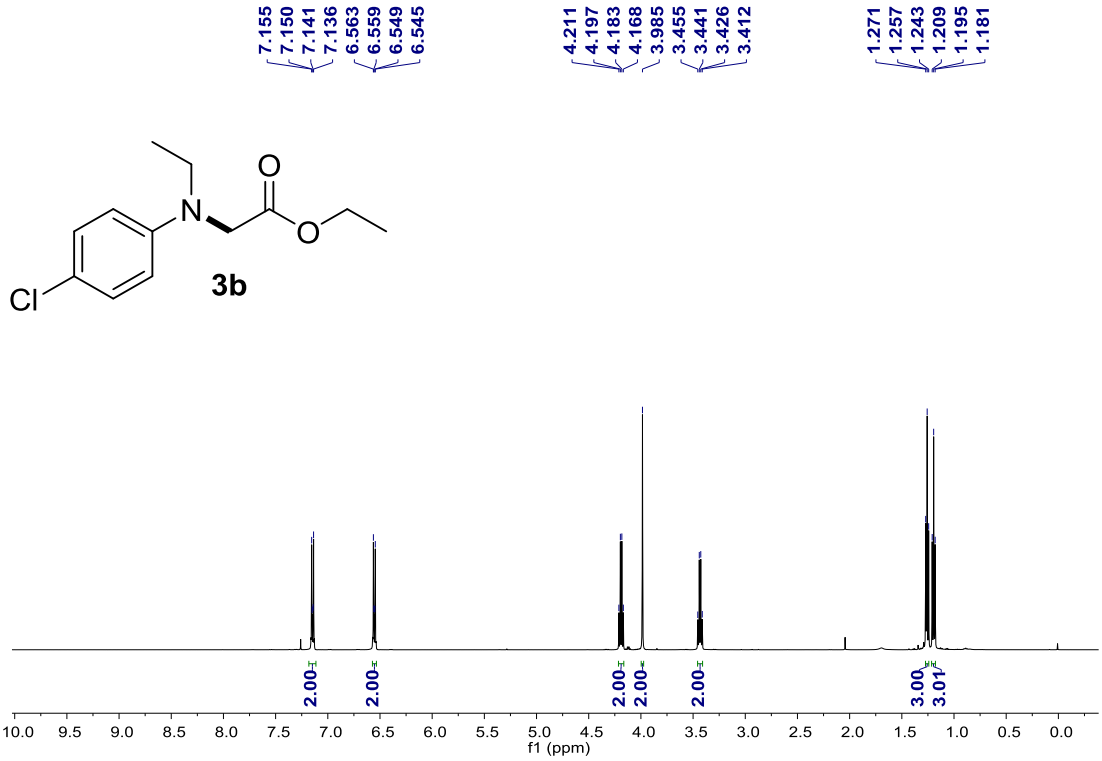
Ⅳ. Copies of ^{1}H and ^{13}C NMR Spectra

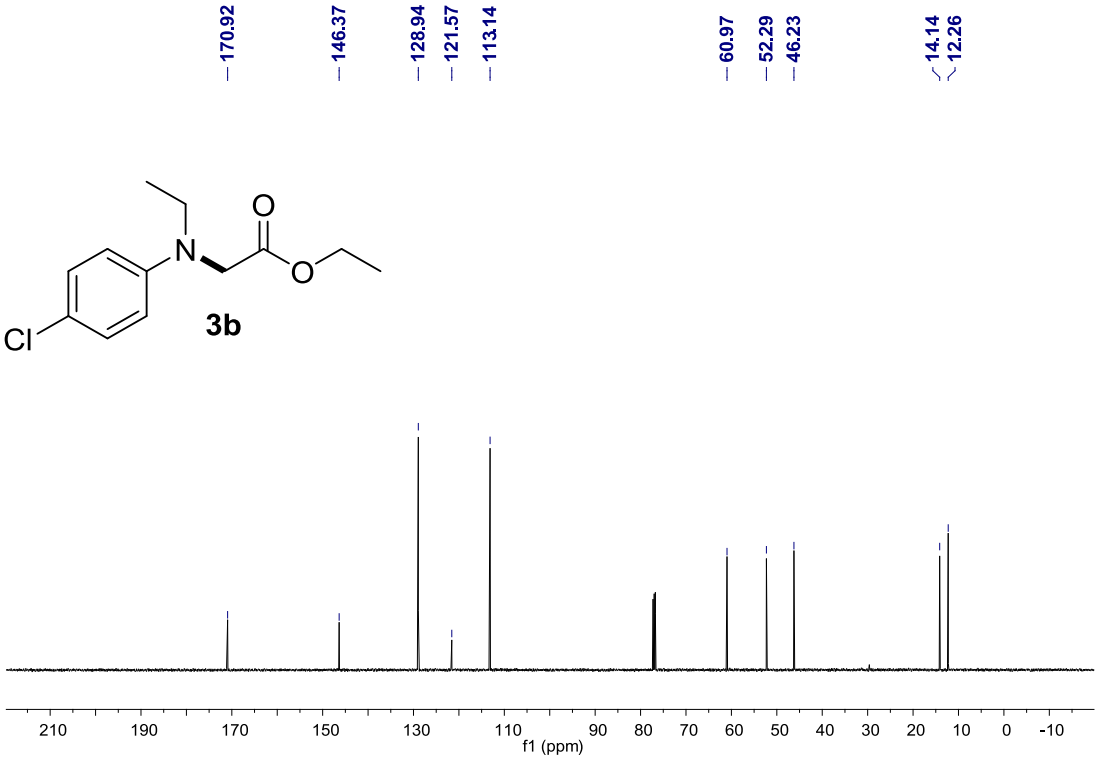


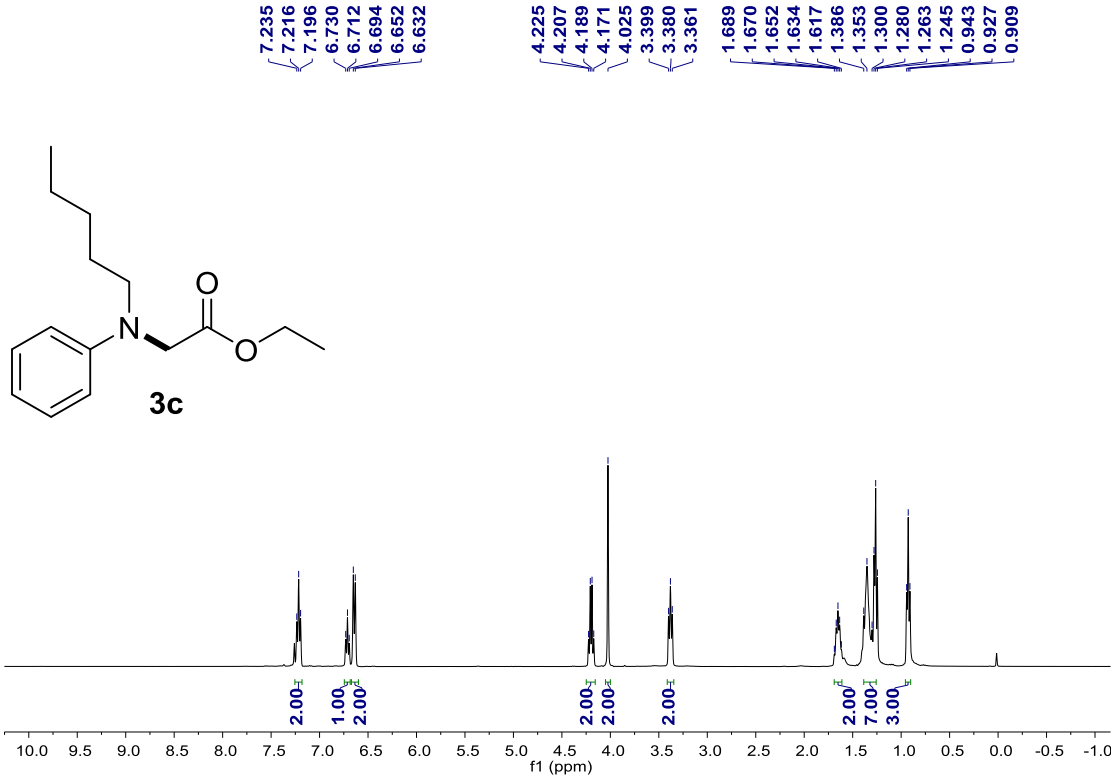







7.155
7.150
7.141
7.136
6.563
6.559
6.549
6.545
4.211
4.197
4.183
4.168
3.985
3.455
3.441
3.426
3.412
1.271
1.257
1.243
1.209
1.195
1.181
O
N
O
Cl
3b
2.00
2.00
2.00
2.00
2.00
3.00
3.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


170.92
146.37
128.94
121.57
113.14
60.97
52.29
46.23
14.14
12.26
O
N
O
Cl
3b
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.235
7.216
7.196
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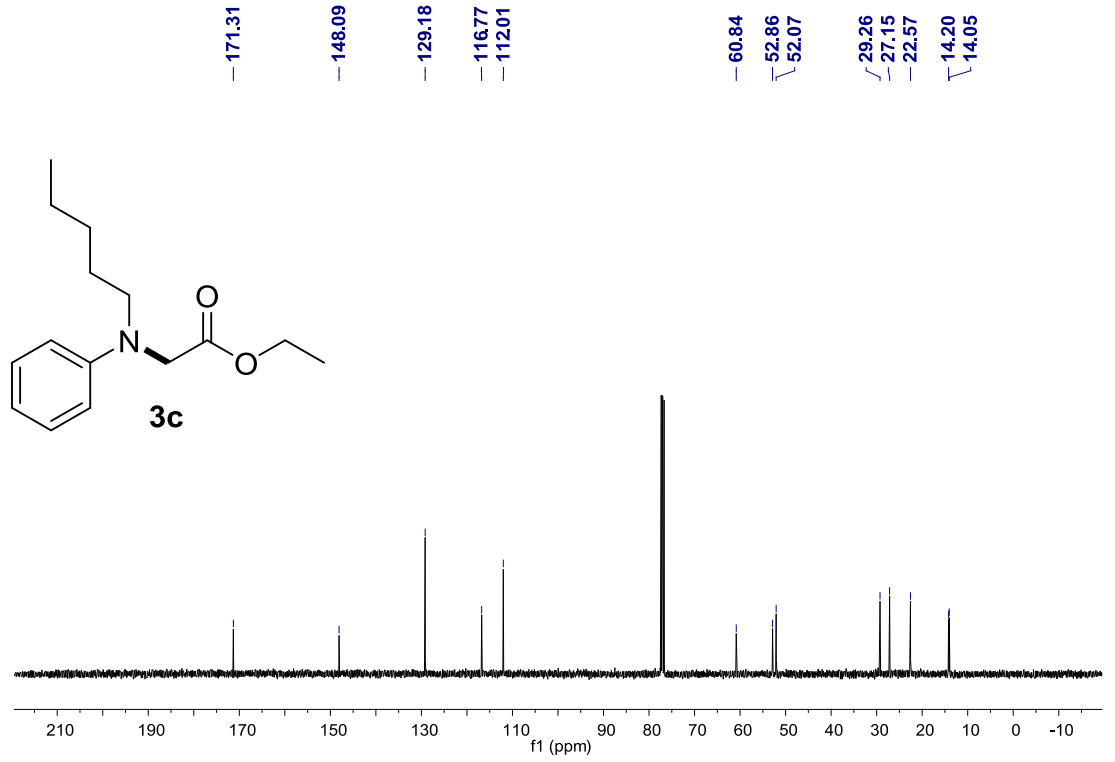
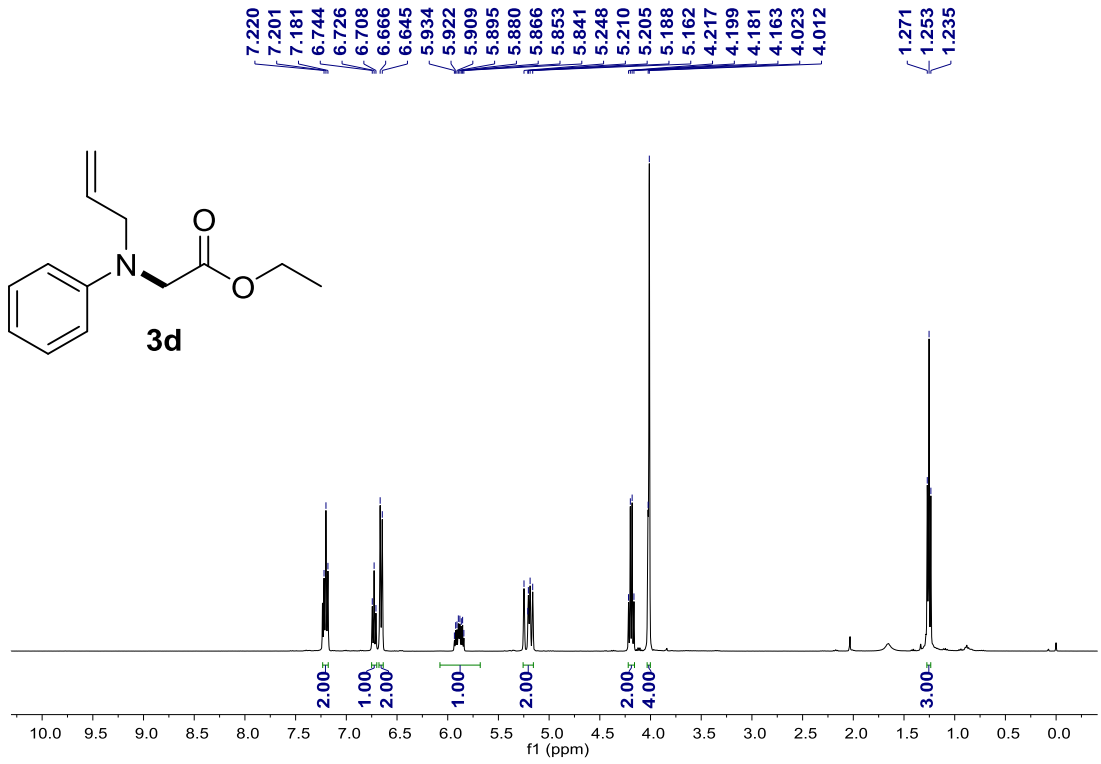
6.712
6.694
6.652
6.632
4.225
4.207
4.189
4.171
4.025
3.399
3.380
3.361
1.689
1.670
1.652
1.634
1.617
1.386
1.353
1.300
1.280
1.263
1.245
0.943
0.927
0.909
O
N
O
3c
2.00
1.00
2.00
2.00
2.00
2.00
2.00
7.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


171.31
148.09
129.18
116.77
112.01
60.84
52.86
52.07
29.26
27.15
22.57
14.20
14.05
O
N
O
3c
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.220
7.201
7.181
6.744
6.726
6.708
6.666
6.645
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5.922
5.909
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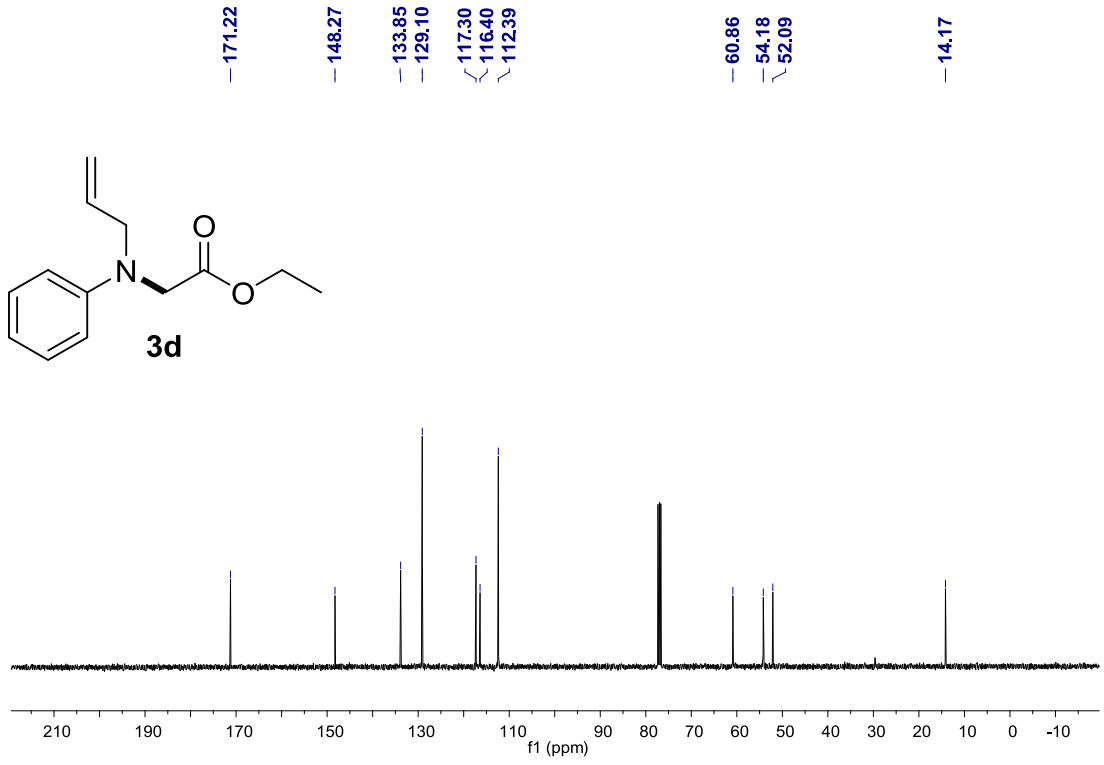
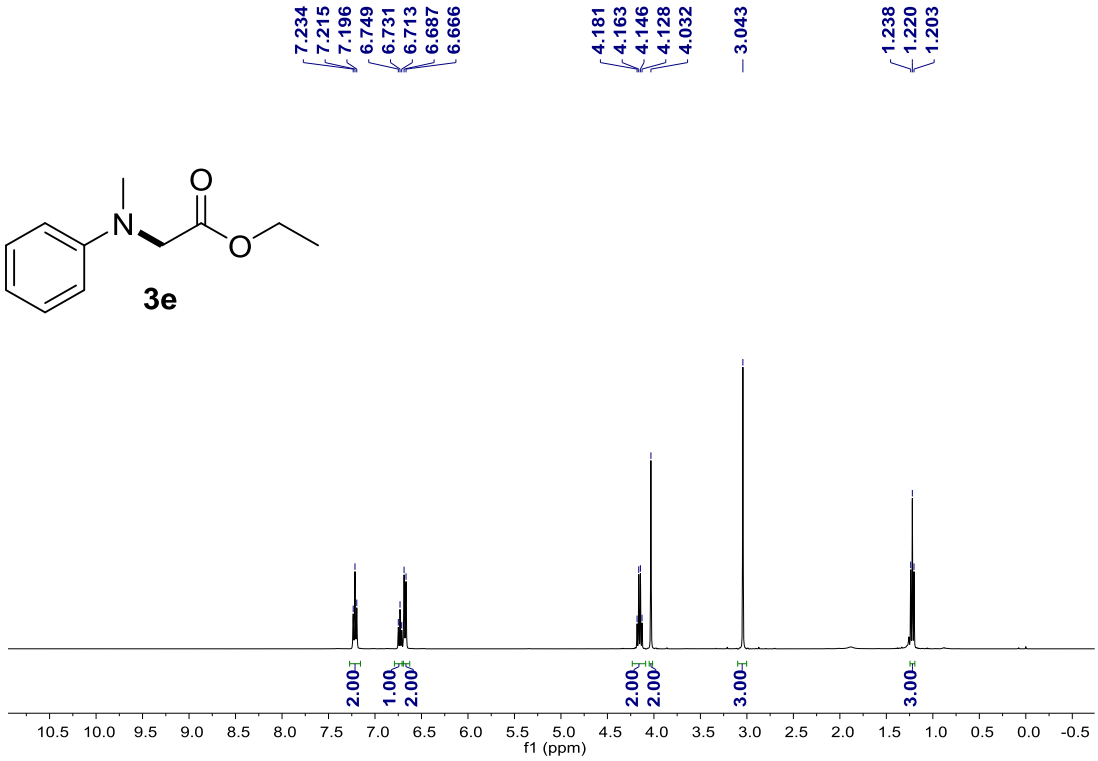
5.880
5.866
5.853
5.841
5.248
5.210
5.205
5.188
5.162
4.217
4.199
4.181
4.163
4.023
4.012
1.271
1.253
1.235
O
N
O
3d
2.00
1.00
2.00
1.00
2.00
2.00
4.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


171.22
148.27
133.85
129.10
117.30
116.40
112.39
60.86
54.18
52.09
14.17
O
N
O
3d
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.234
7.215
7.196
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6.731
6.713
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6.666
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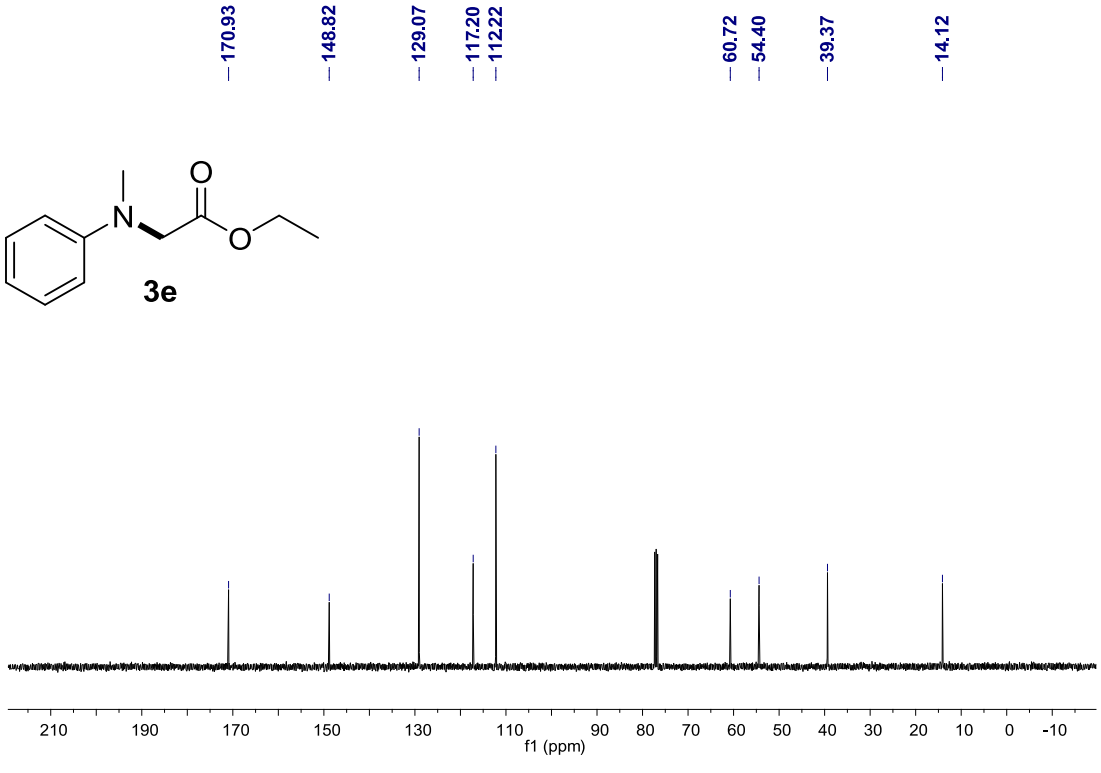
4.163
4.146
4.128
4.032
3.043
1.238
1.220
1.203
O
N
O
3e
2.00
1.00
2.00
2.00
2.00
3.00
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


170.93
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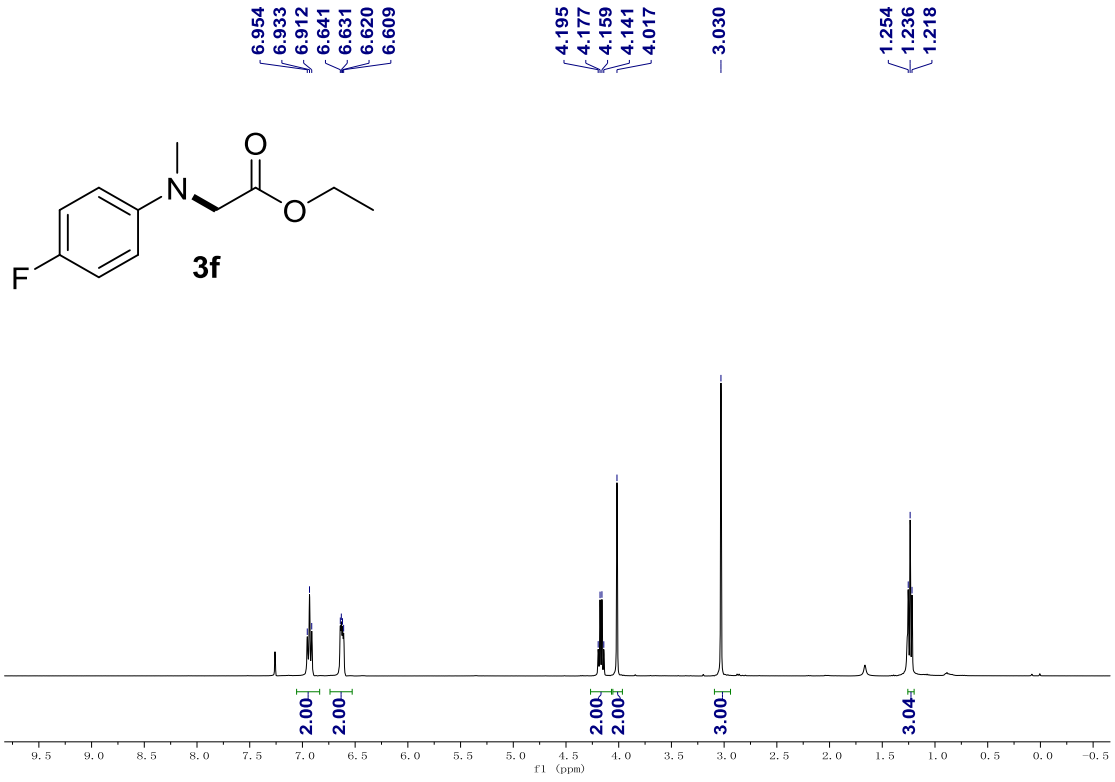
129.07
117.20
11222
60.72
54.40
39.37
14.12
O
N
O
3e
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


6.954
6.933
6.912
6.641
6.631
6.620
6.609
4.195
4.177
4.159
4.141
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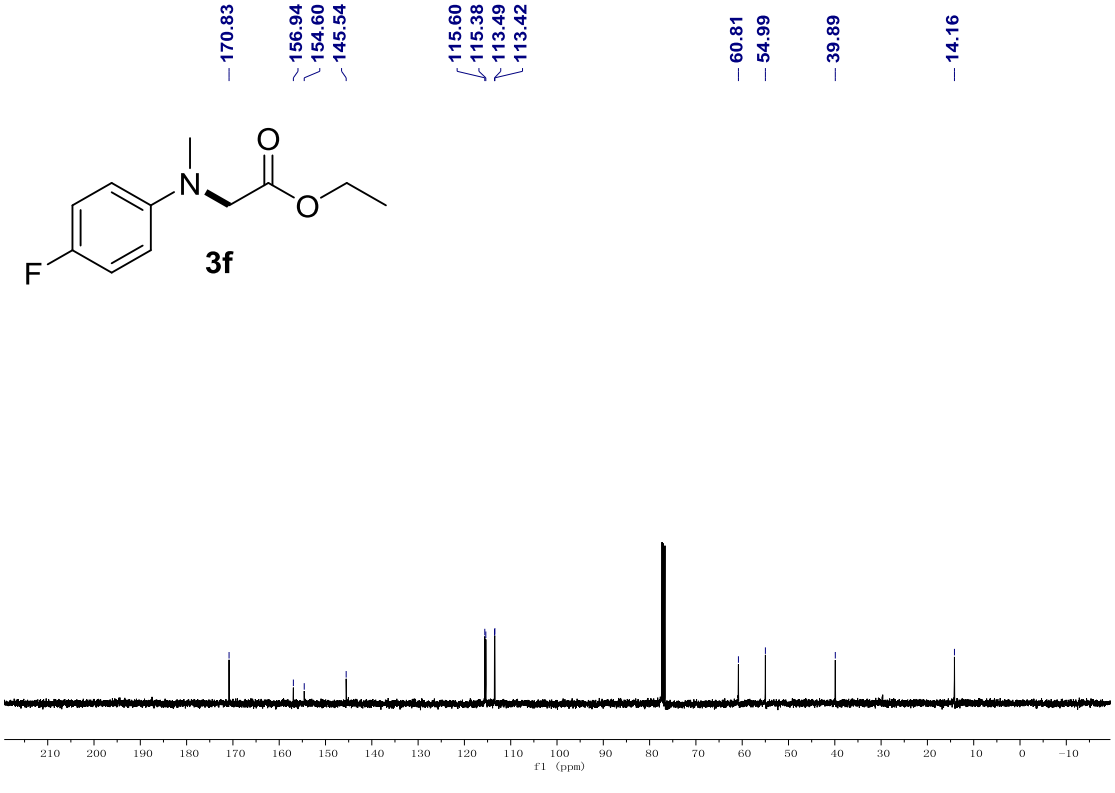
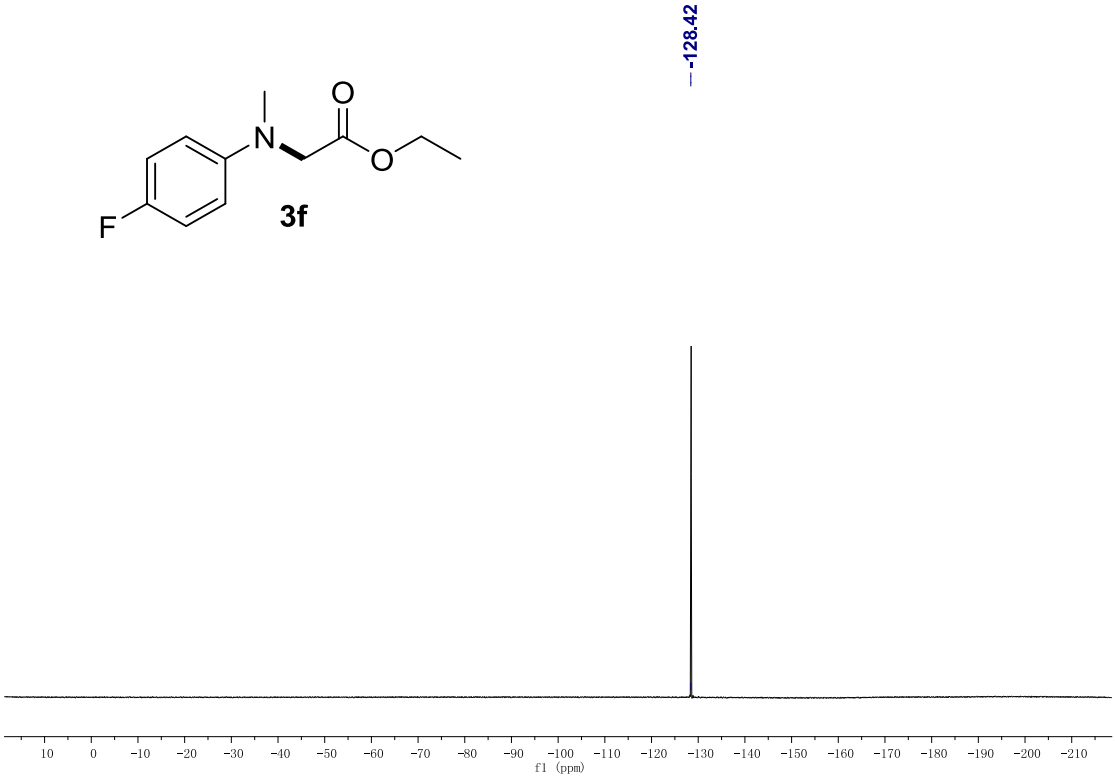
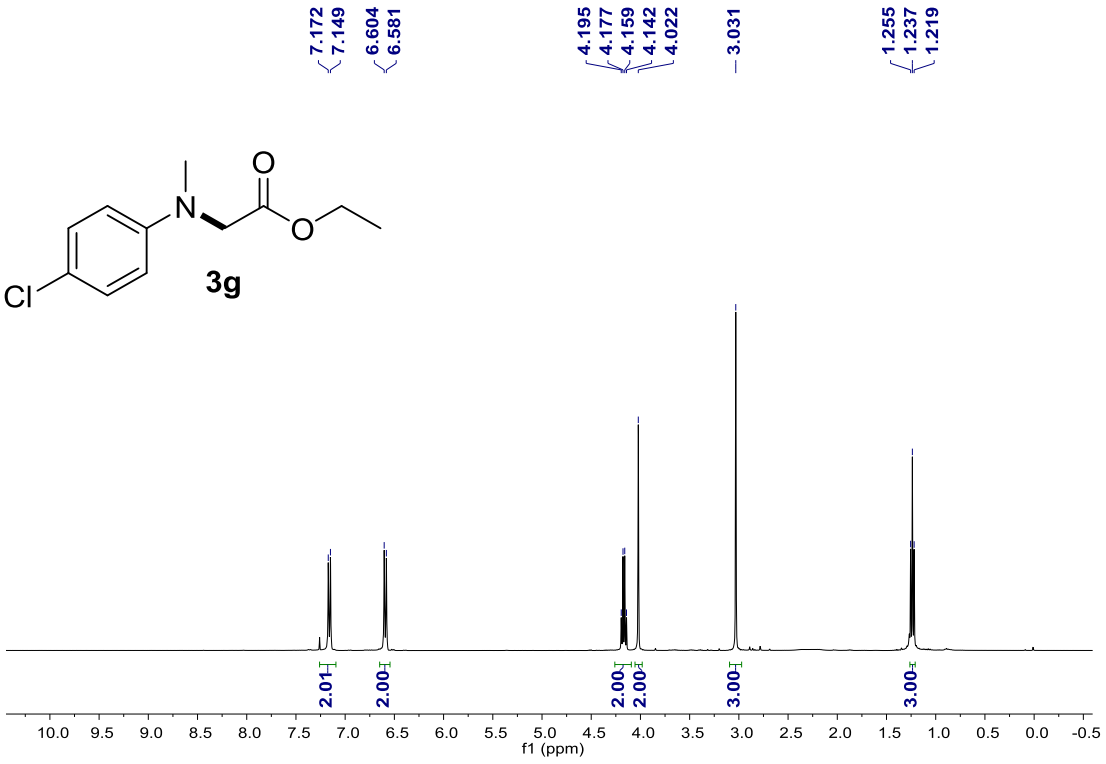
3.030
1.254
1.236
1.218
O
N
O
F
3f
2.00
2.00
2.00
2.00
3.00
3.04
f1 (ppm)

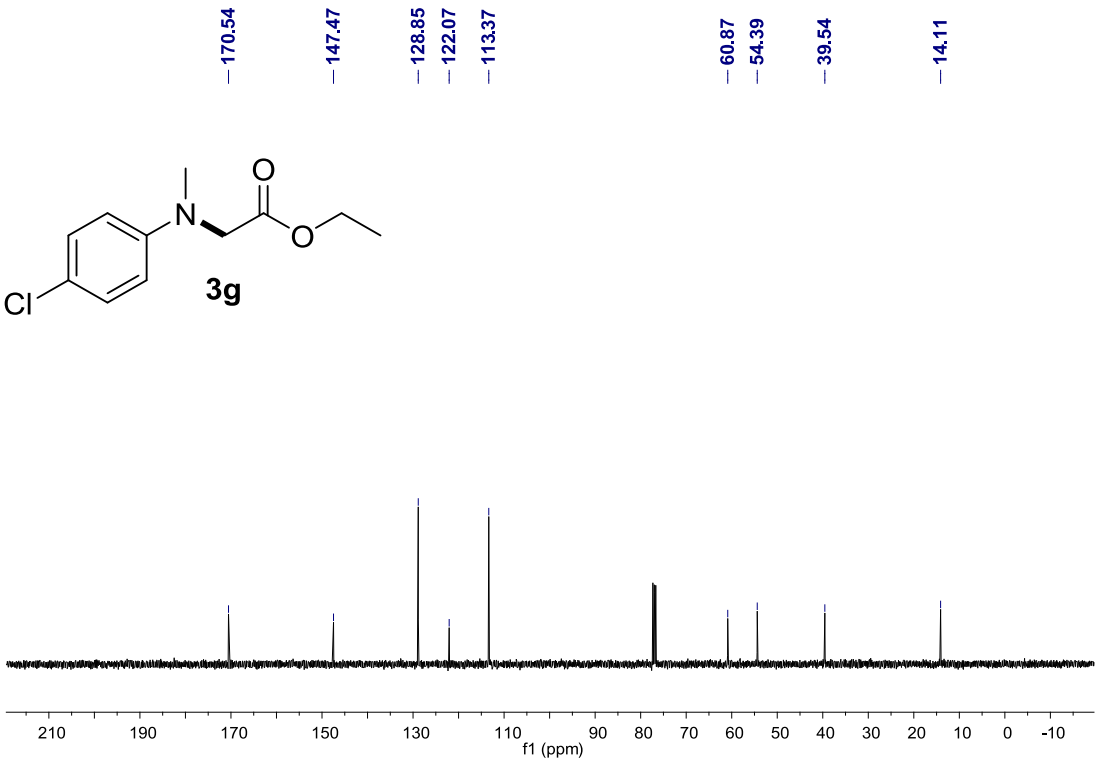

170.83
156.94
154.60
145.54
115.60
115.38
113.49
113.42
60.81
54.99
39.89
14.16
O
N
O
F
3f
f1 (ppm)

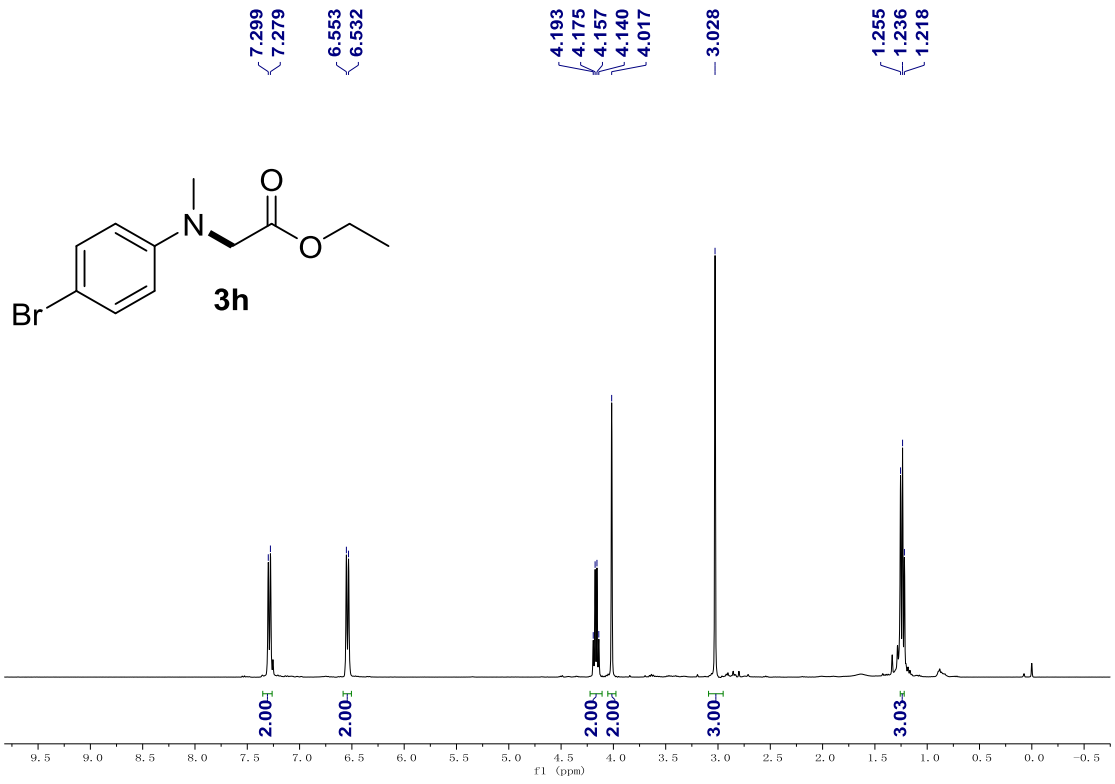
O
N
O
F

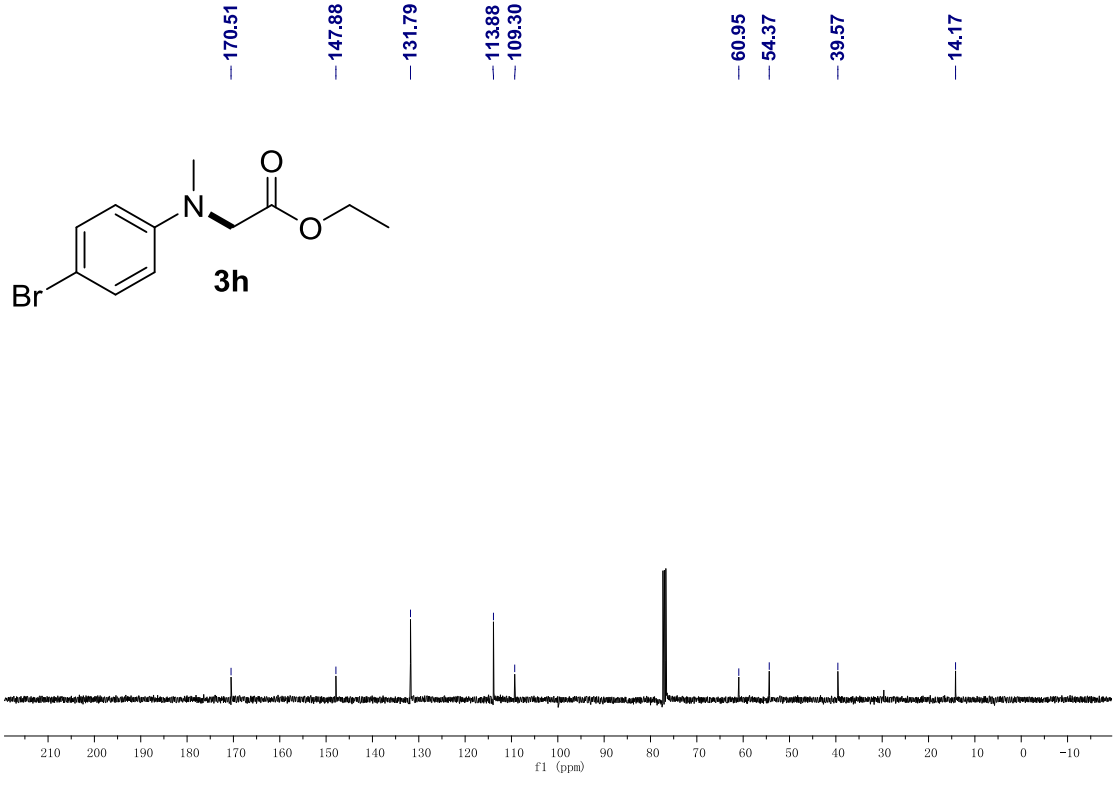
3f

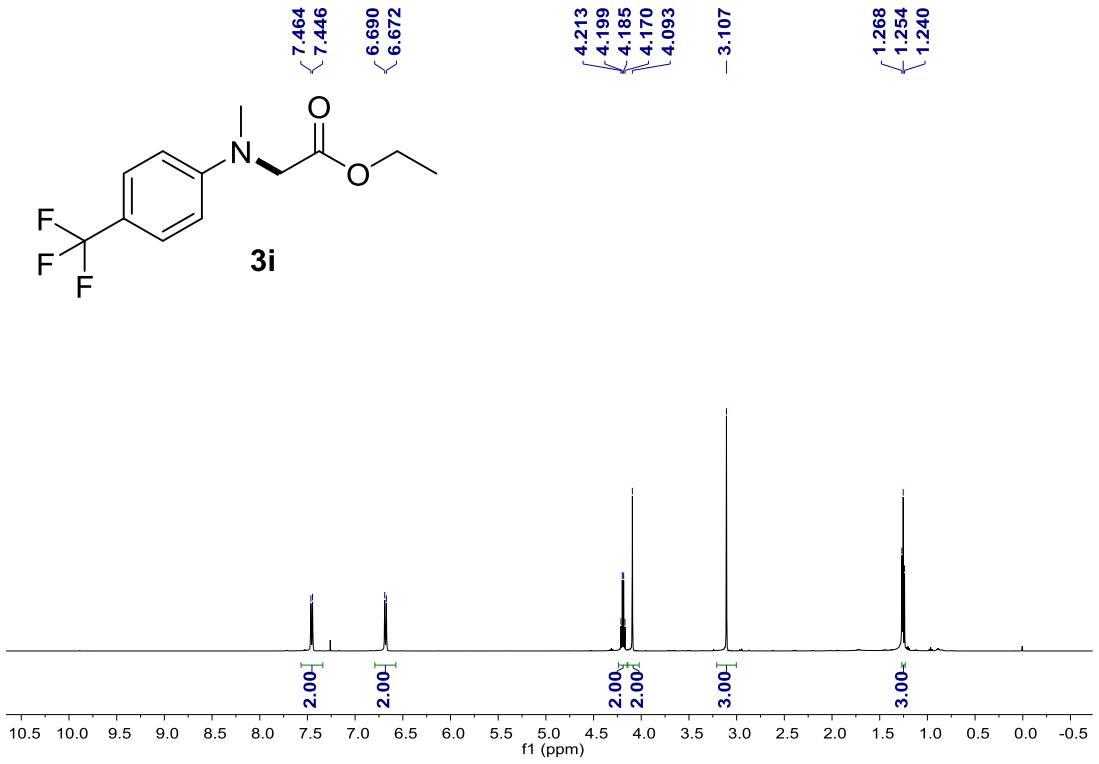

-128.42
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

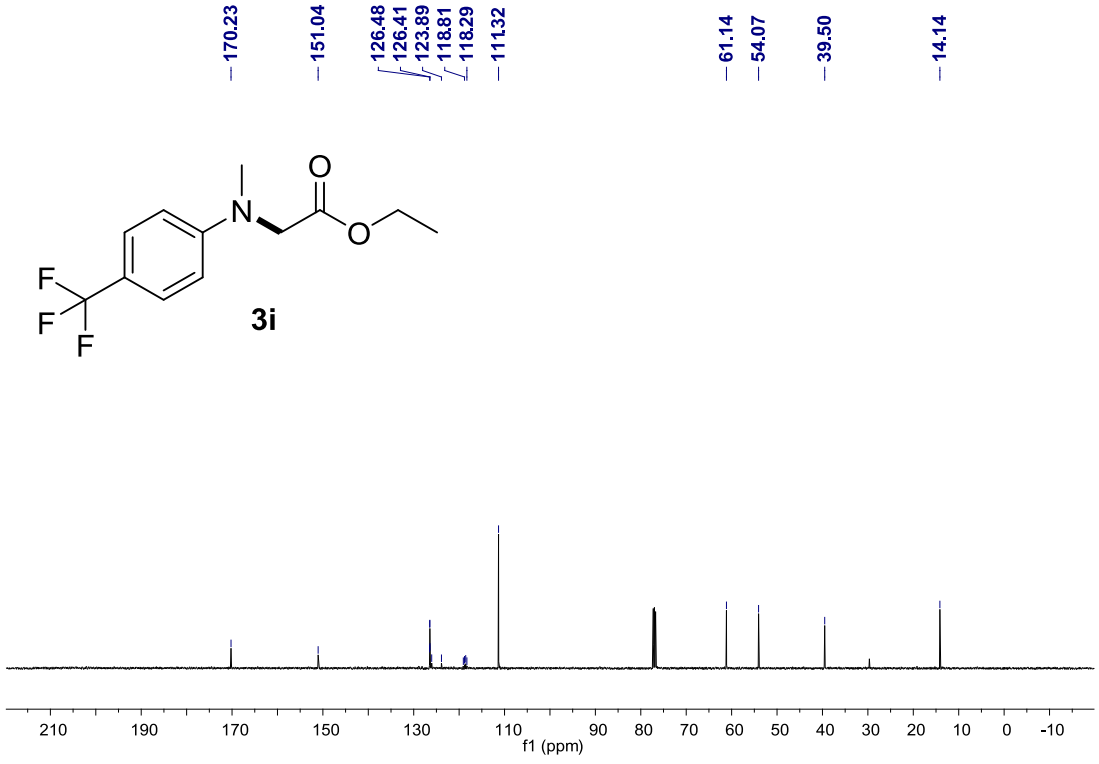

7.172
7.149
6.604
6.581
4.195
4.177
4.159
4.142
4.022
3.031
1.255
1.237
1.219
Cl
N
O
O
3g
2.01
2.00
2.00
2.00
3.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

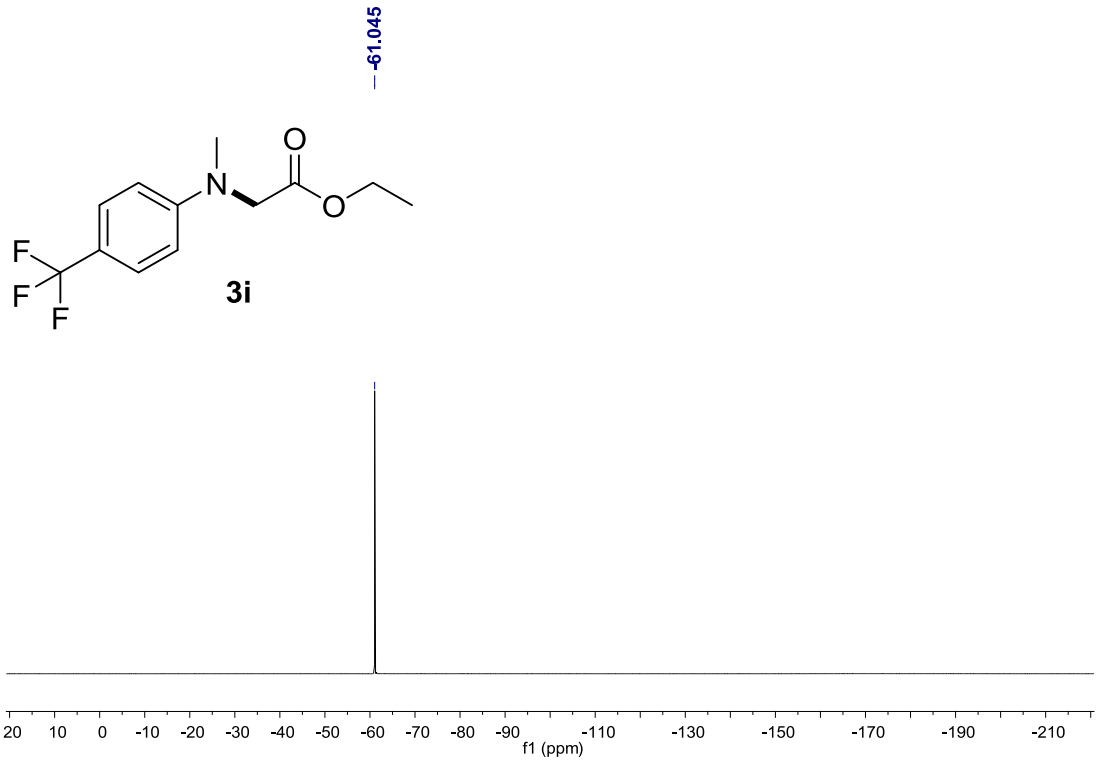

170.54
147.47
128.85
122.07
113.37
60.87
54.39
39.54
14.11
Cl
N
O
O
3g
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

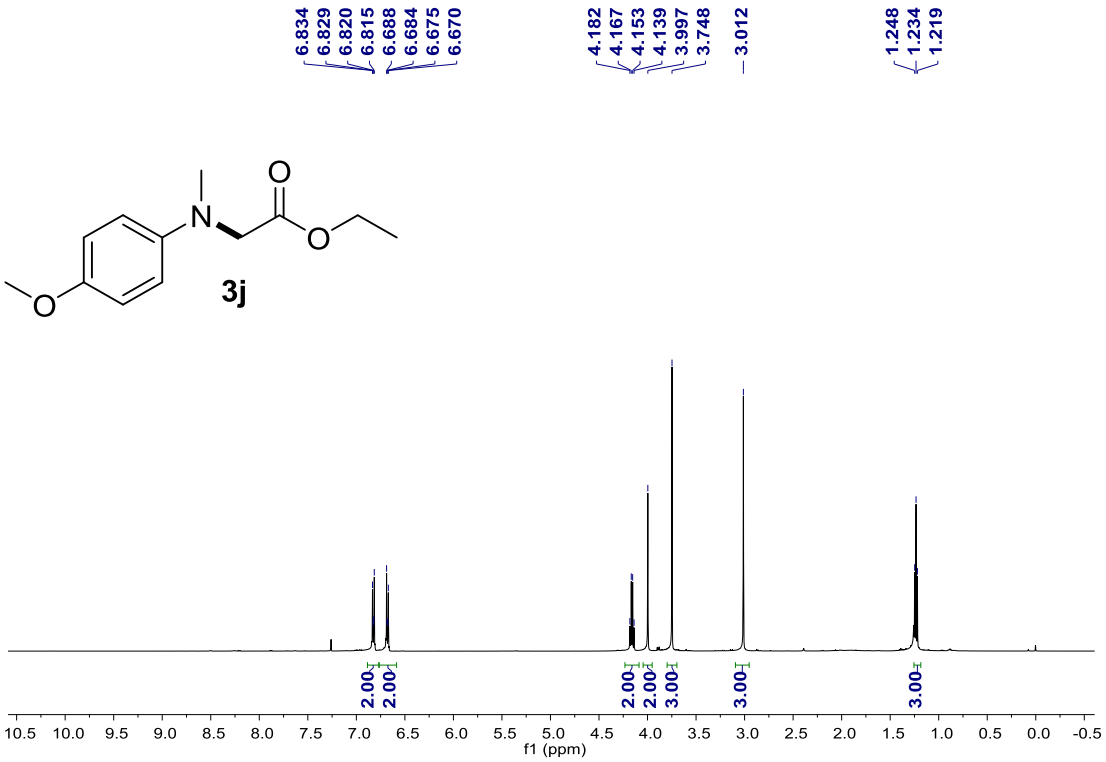

7.299
7.279
6.553
6.532
4.193
4.175
4.157
4.140
4.017
3.028
1.255
1.236
1.218
Br
N
O
O
3h
2.00
2.00
2.00
2.00
3.00
3.03
f1 (ppm)

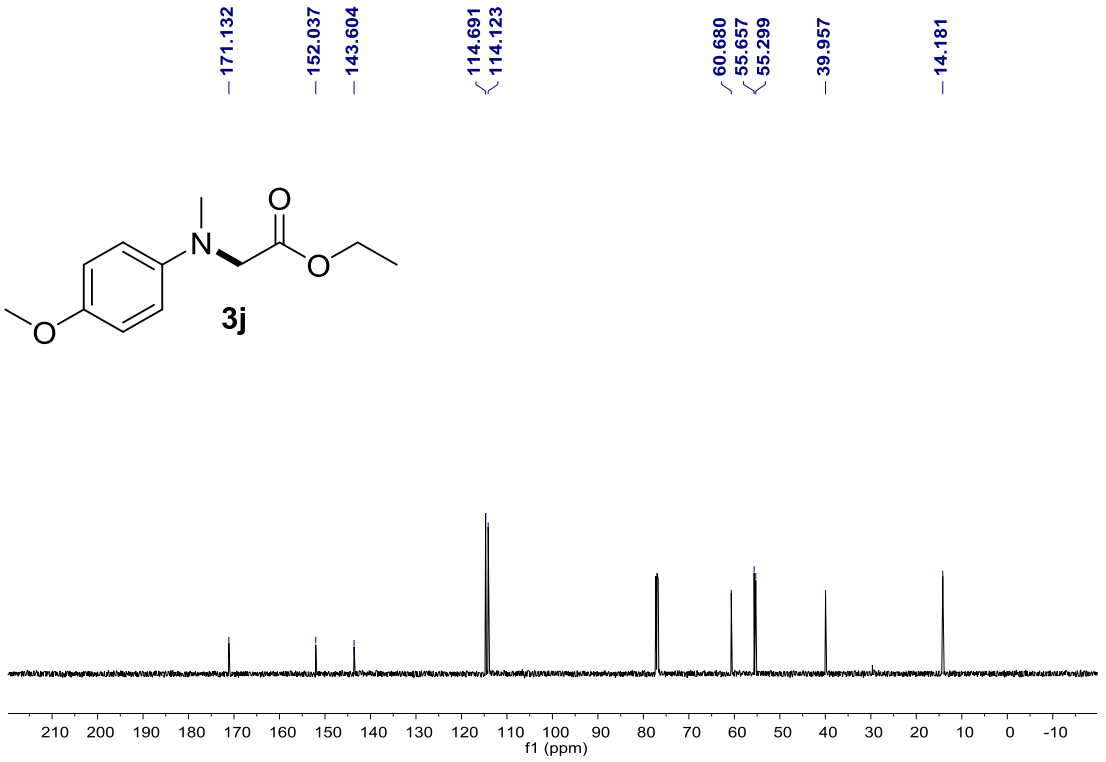

170.51
147.88
131.79
113.88
109.30
60.95
54.37
39.57
14.17
Br
N
O
O
3h
f1 (ppm)

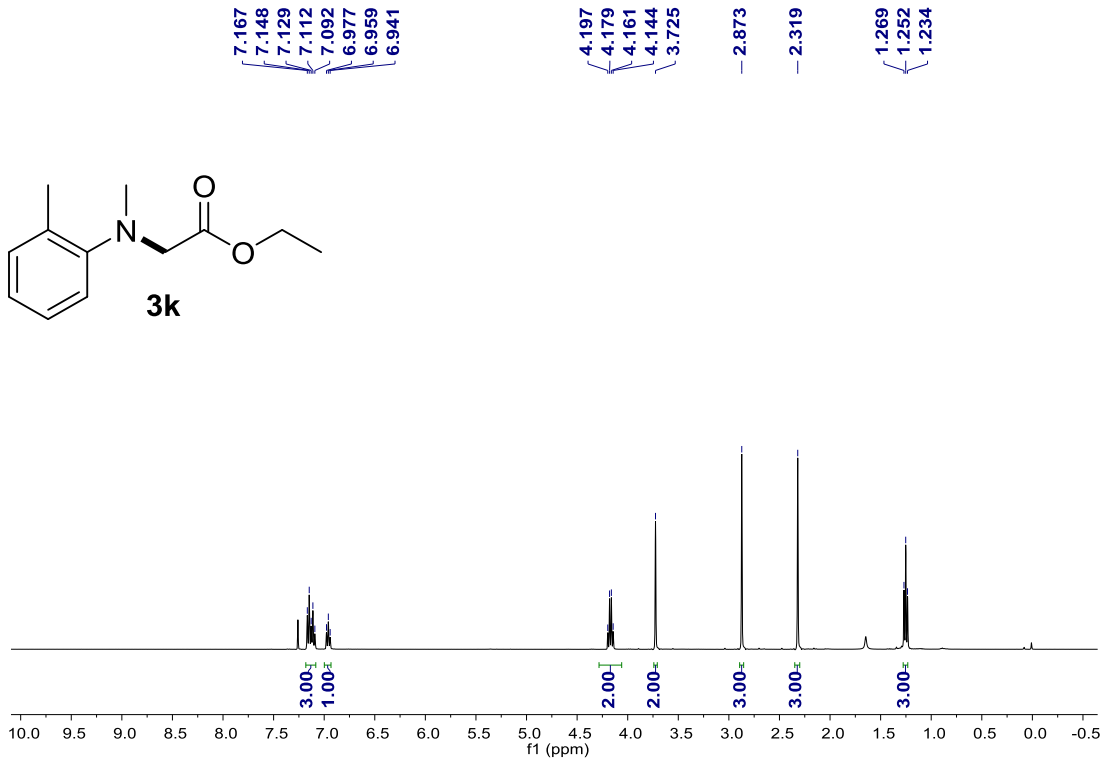

7.464
7.446
6.690
6.672
4.213
4.199
4.185
4.170
4.093
3.107
1.268
1.254
1.240
O
N
O
F
F
F
3i
2.00
2.00
2.00
2.00
3.00
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

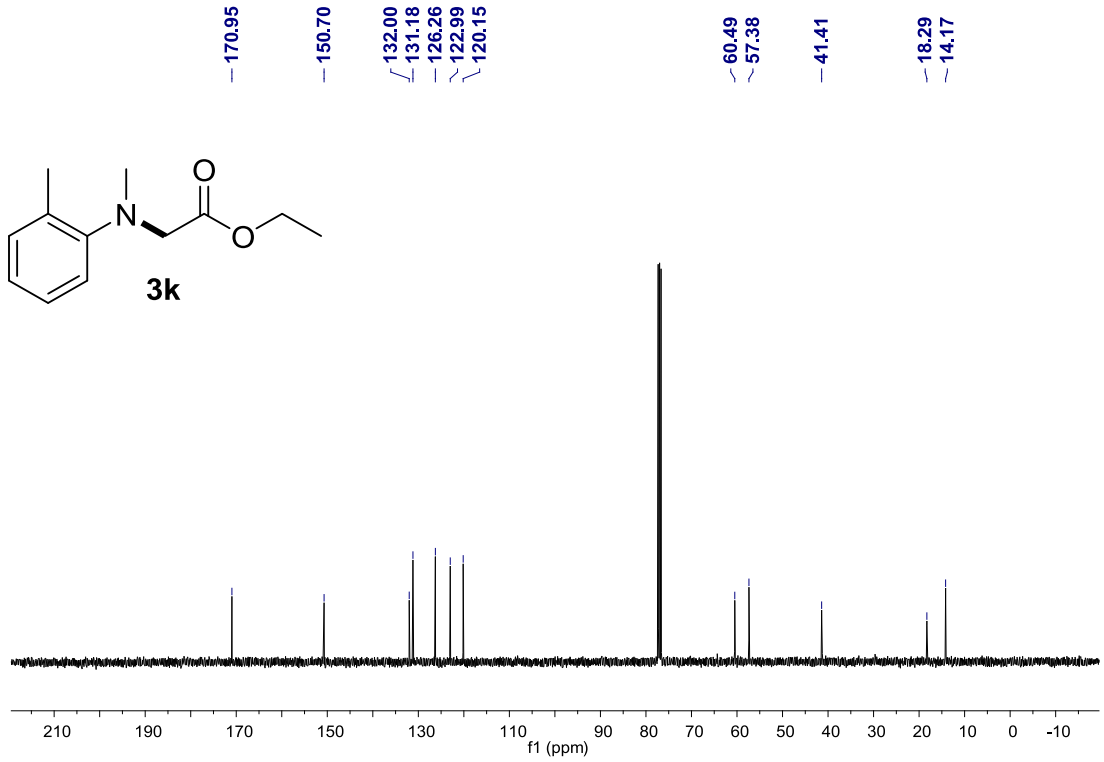

170.23
151.04
126.48
126.41
123.89
118.81
118.29
111.32
61.14
54.07
39.50
14.14
O
N
O
F
F
F
3i
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

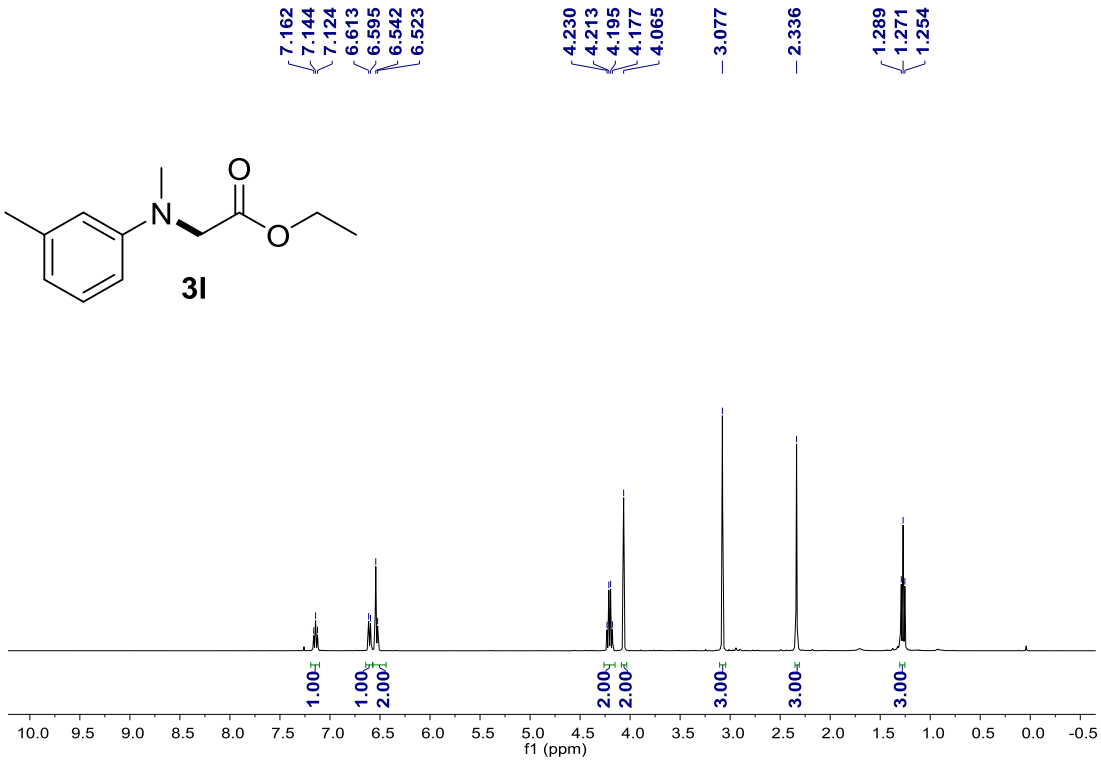

-61.045
O
N
O
F
F
F
3i
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

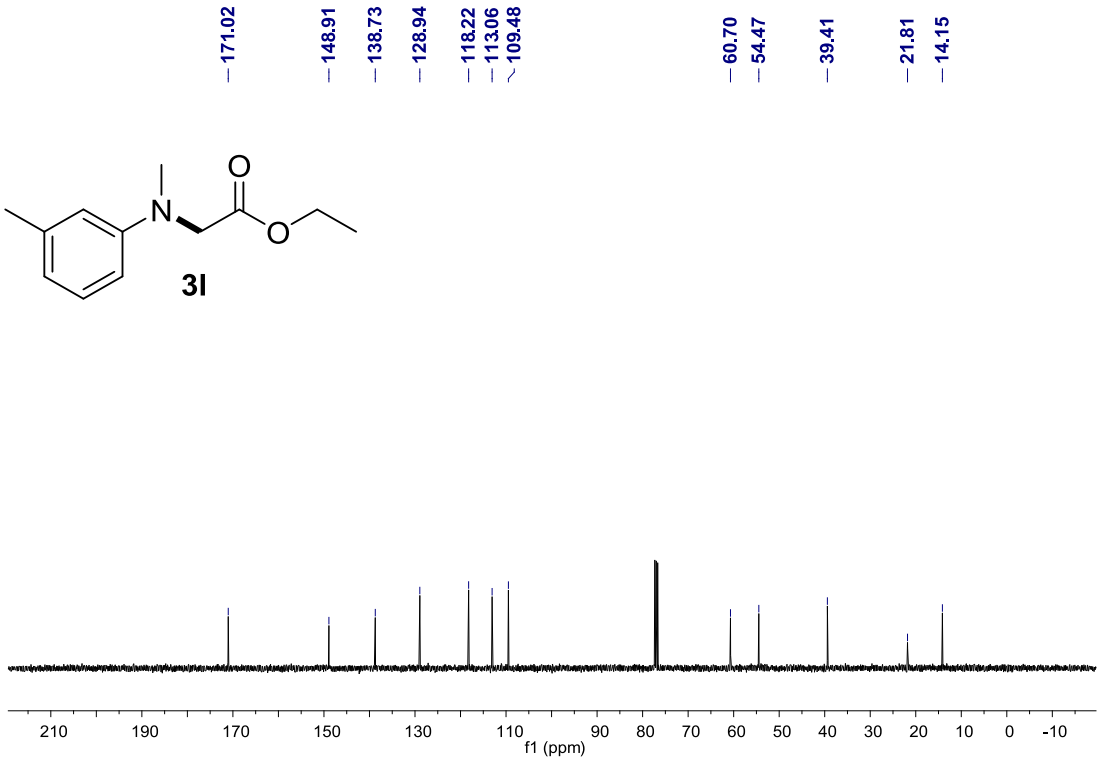

6.834
6.829
6.820
6.815
6.688
6.684
6.675
6.670
4.182
4.167
4.153
4.139
3.997
3.748
3.012
1.248
1.234
1.219
3j
2.00
2.00
2.00
2.00
3.00
3.00
3.00
f1 (ppm)

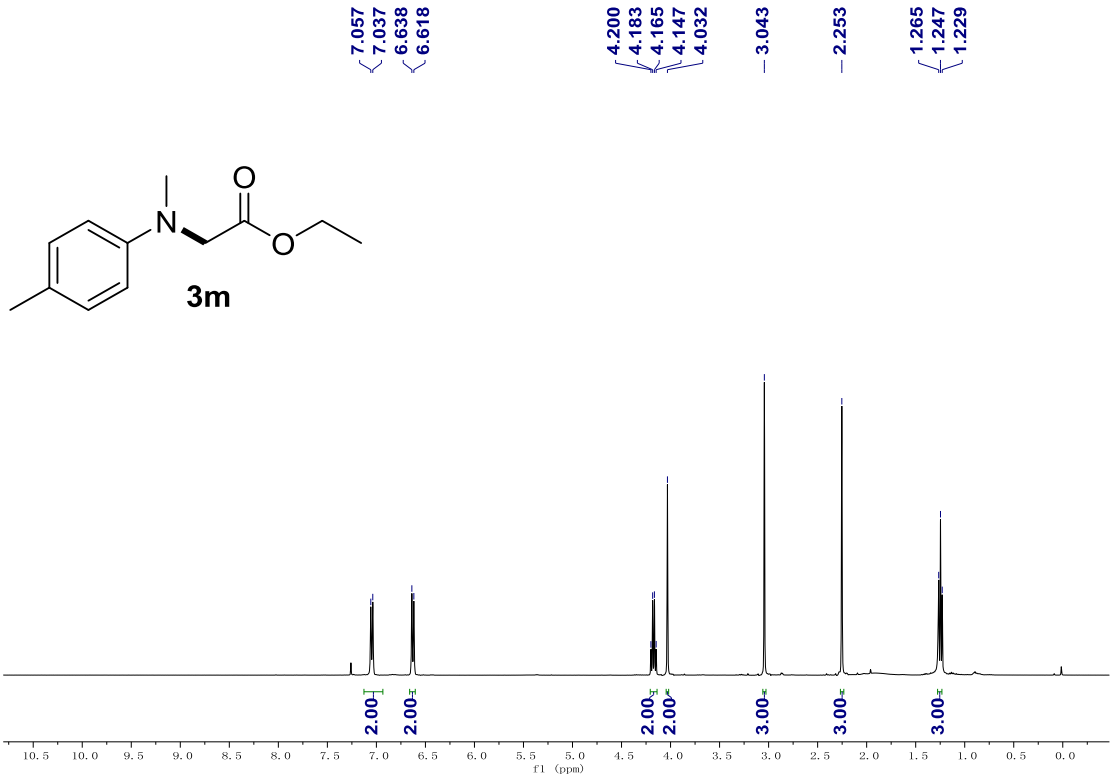

171.132
152.037
143.604
114.691
114.123
60.680
55.657
55.299
39.957
14.181
3j
f1 (ppm)

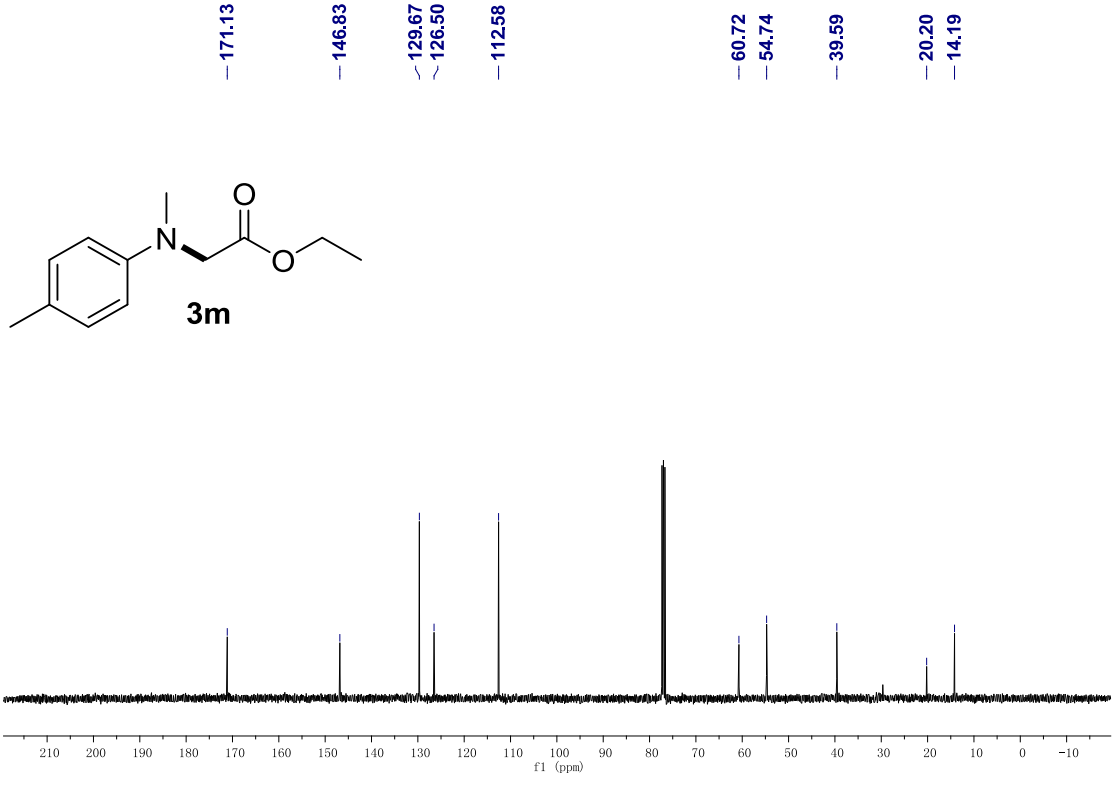

7.167
7.148
7.129
7.112
7.092
6.977
6.959
6.941
4.197
4.179
4.161
4.144
3.725
2.873
2.319
1.269
1.252
1.234
O
N
O
3k
3.00
1.00
2.00
2.00
3.00
3.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

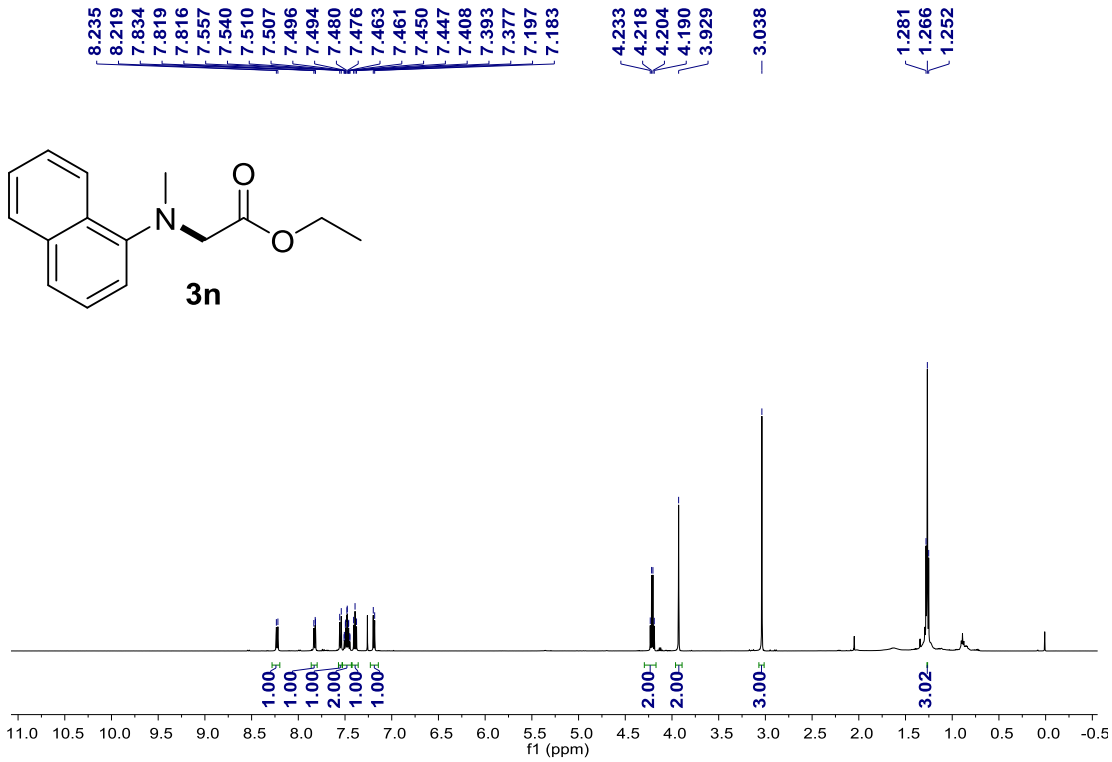

170.95
150.70
132.00
131.18
126.26
122.99
120.15
60.49
57.38
41.41
18.29
14.17
O
N
O
3k
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.162
7.144
7.124
6.613
6.595
6.542
6.523
4.230
4.213
4.195
4.177
4.065
3.077
2.336
1.289
1.271
1.254
O
N
O
3l
1.00
1.00
2.00
2.00
2.00
3.00
3.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


171.02
148.91
138.73
128.94
118.22
113.06
109.48
60.70
54.47
39.41
21.81
14.15
O
N
O
3l
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.057
7.037
6.638
6.618
4.200
4.183
4.165
4.147
4.032
3.043
2.253
1.265
1.247
1.229
3m
2.00
2.00
2.00
2.00
3.00
3.00
3.00
f1 (ppm)


171.13
146.83
129.67
126.50
112.58
60.72
54.74
39.59
20.20
14.19
3m
f1 (ppm)


8.235
8.219
7.834
7.819
7.816
7.557
7.540
7.510
7.507
7.496
7.494
7.480
7.476
7.463
7.461
7.450
7.447
7.408
7.393
7.377
7.197
7.183
4.233
4.218
4.204
4.190
3.929
3.038
1.281
1.266
1.252
3n
1.00
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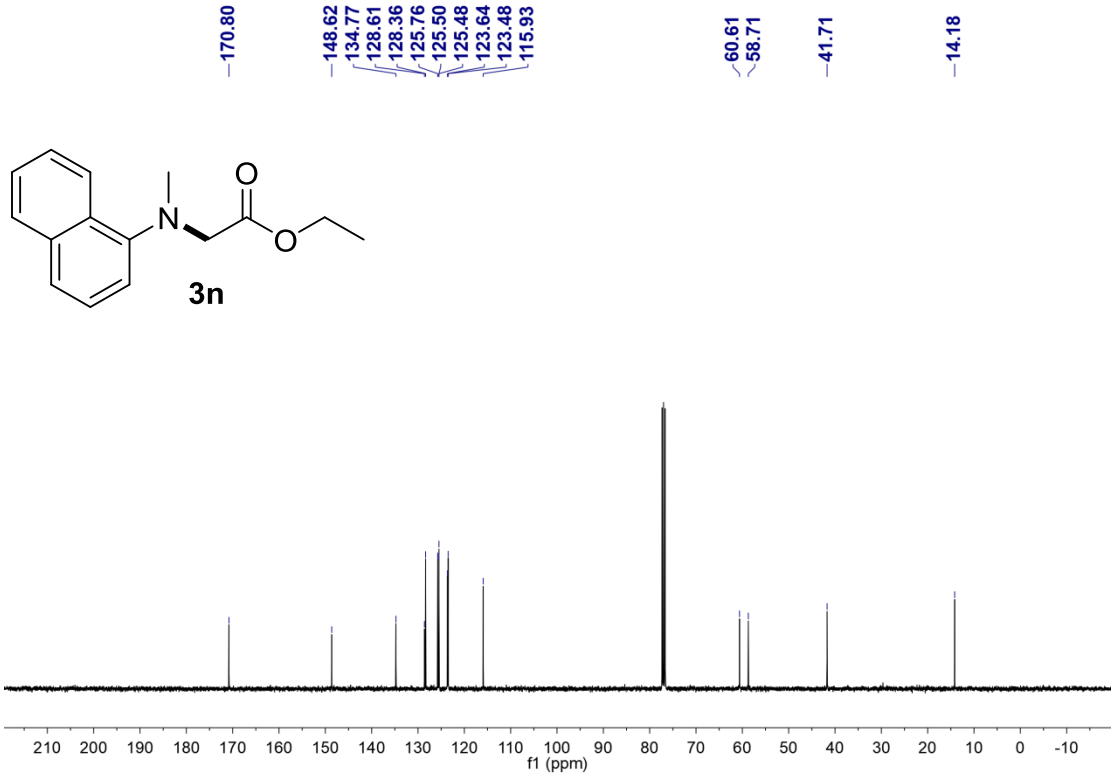
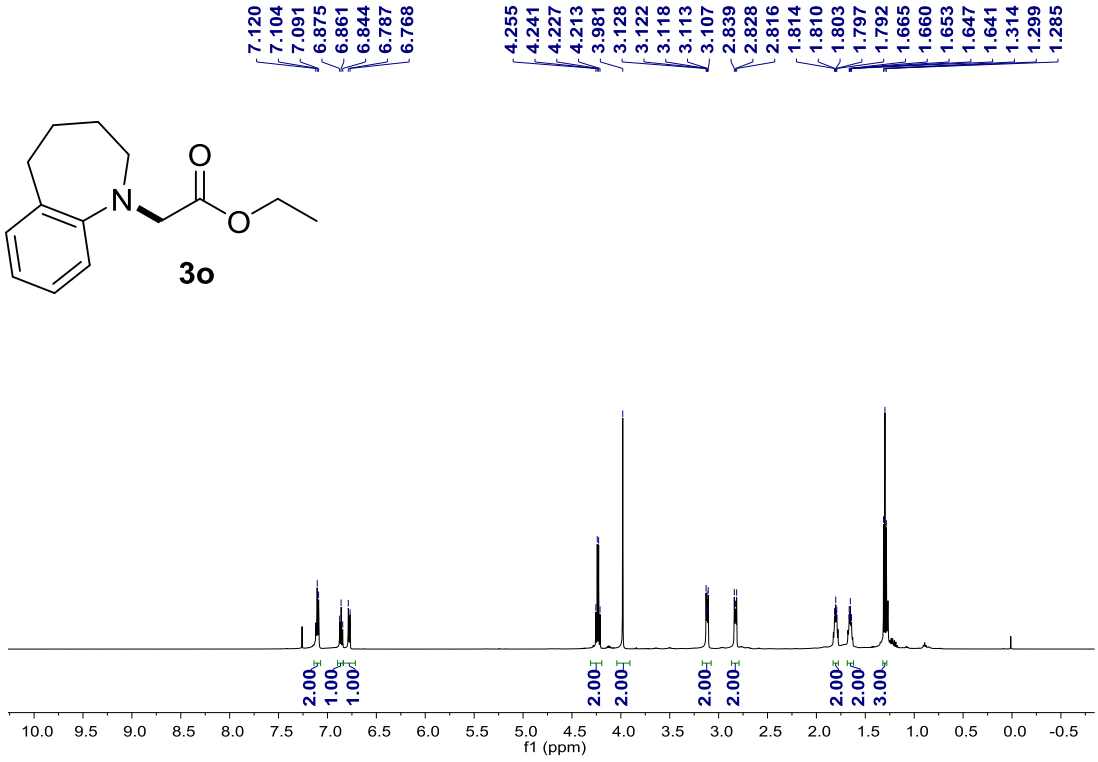
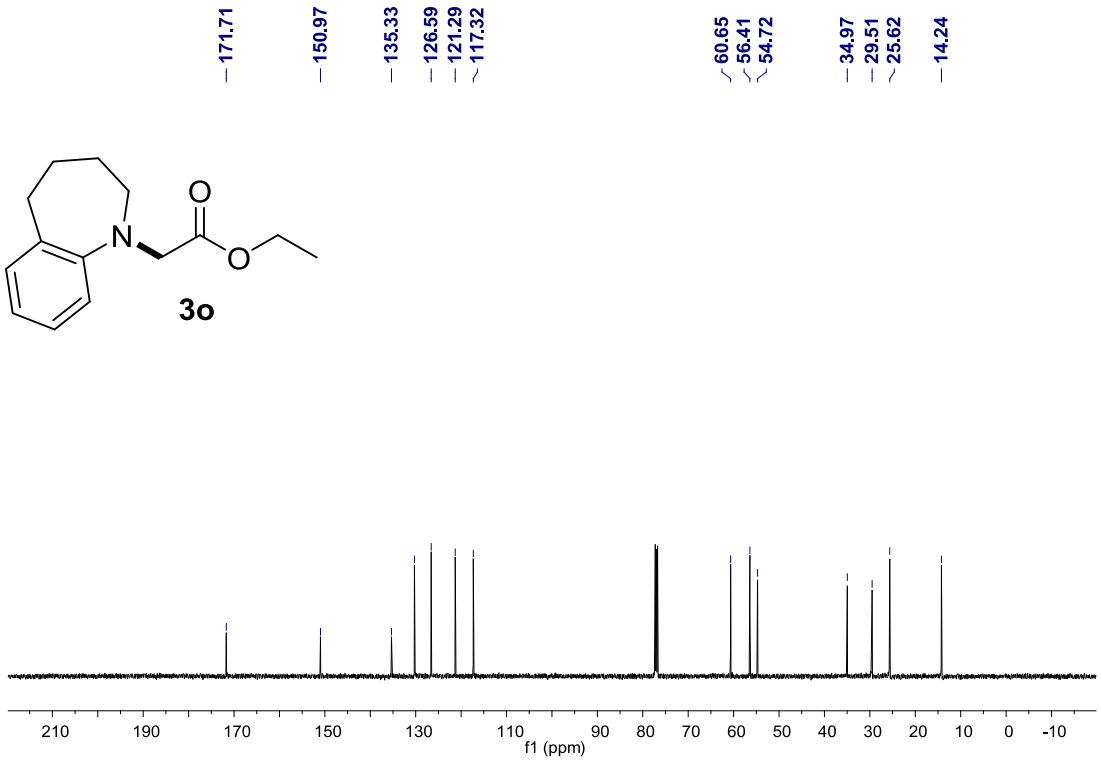
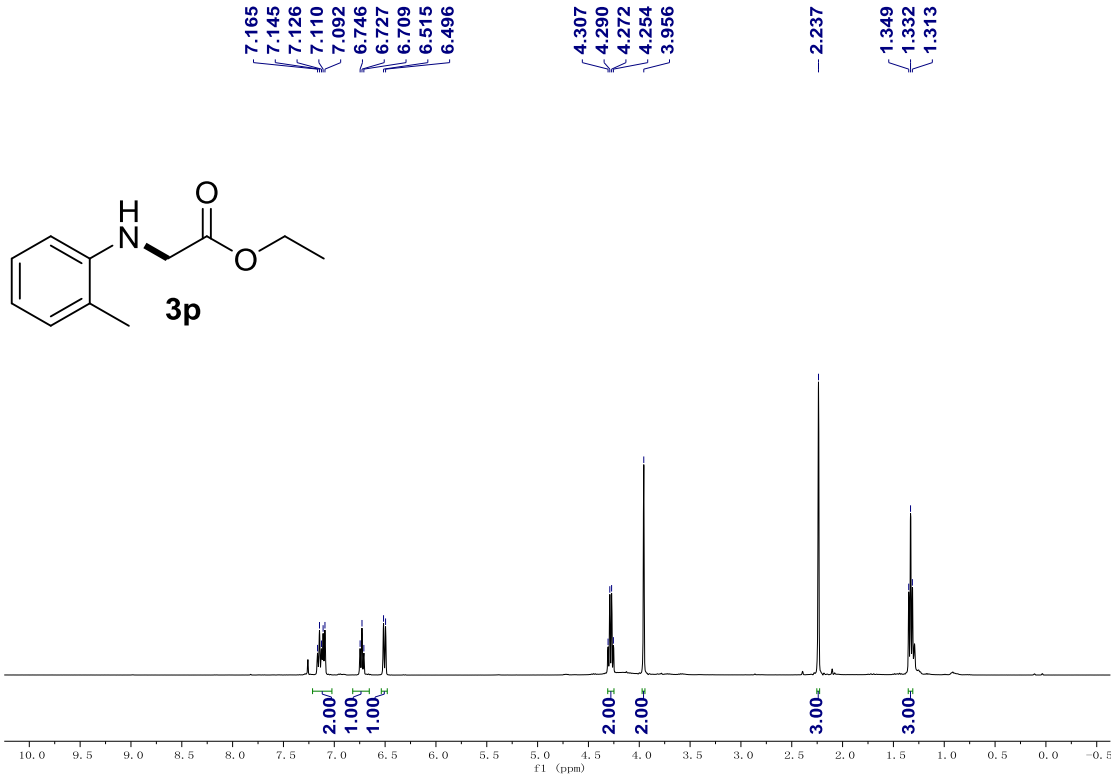
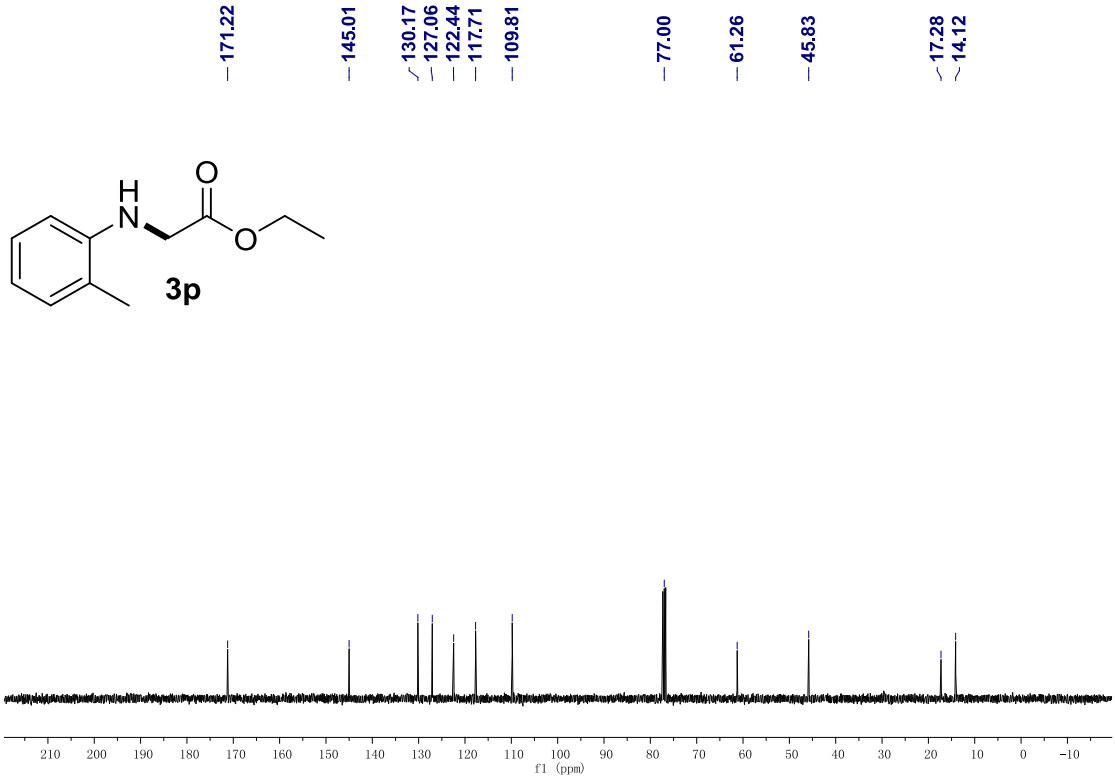
2.00
2.00
3.00
3.02
f1 (ppm)

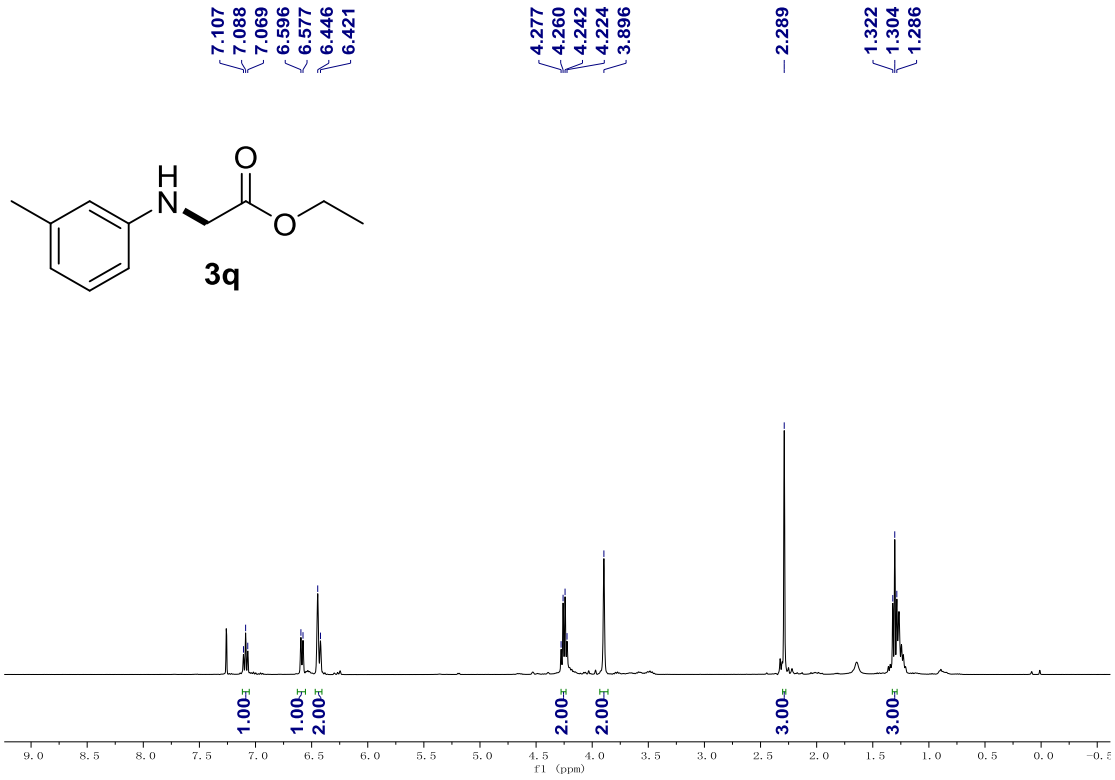

170.80
148.62
134.77
128.61
128.36
125.76
125.50
125.48
123.64
123.48
115.93
60.61
58.71
41.71
14.18
3n
f1 (ppm)


7.120
7.104
7.091
6.875
6.861
6.844
6.787
6.768
4.255
4.241
4.227
4.213
3.981
3.128
3.122
3.118
3.113
3.107
2.839
2.828
2.816
1.814
1.810
1.803
1.797
1.792
1.665
1.660
1.653
1.647
1.641
1.314
1.299
1.285
O
N
O
3o
2.00
1.00
1.00
2.00
2.00
2.00
2.00
2.00
2.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


171.71
150.97
135.33
126.59
121.29
117.32
60.65
56.41
54.72
34.97
29.51
25.62
14.24
O
N
O
3o
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.165
7.145
7.126
7.110
7.092
6.746
6.727
6.709
6.515
6.496
4.307
4.290
4.272
4.254
3.956
2.237
1.349
1.332
1.313
3p
2.00
1.00
1.00
2.00
2.00
3.00
3.00
f1 (ppm)


171.22
145.01
130.17
127.06
122.44
117.71
109.81
77.00
61.26
45.83
17.28
14.12
3p
f1 (ppm)


7.107
7.088
7.069
6.596
6.577
6.446
6.421
4.277
4.260
4.242
4.224
3.896
2.289
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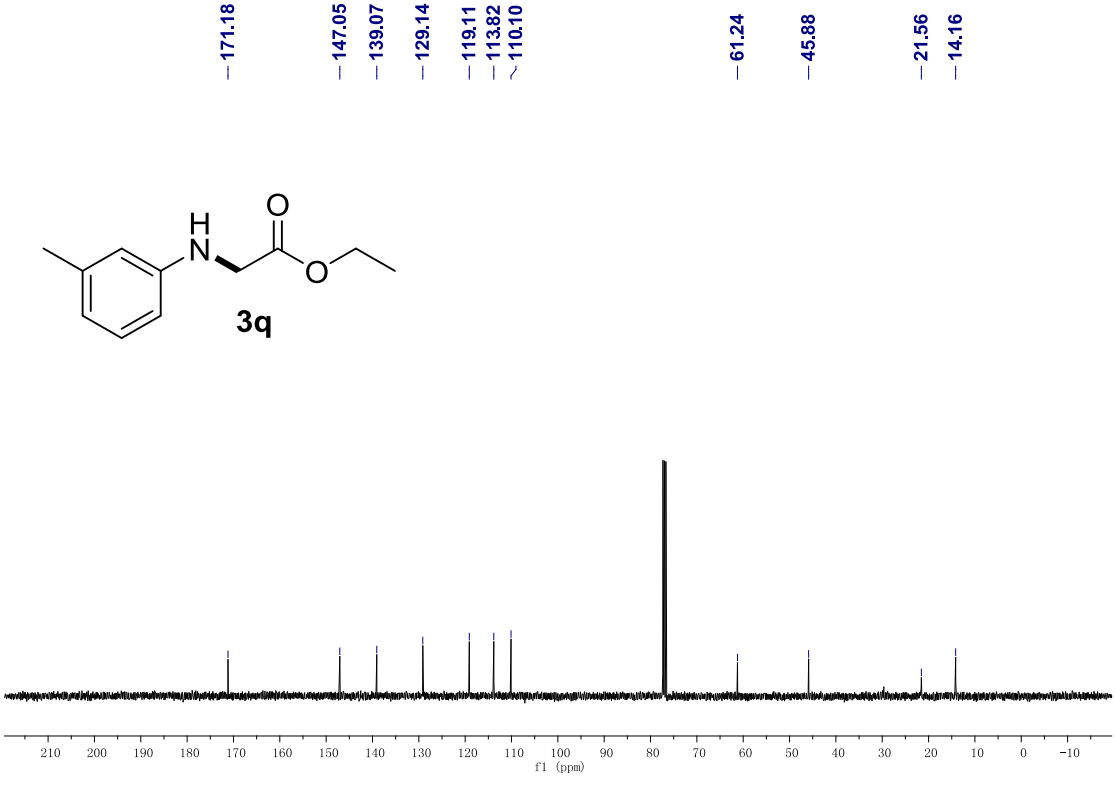
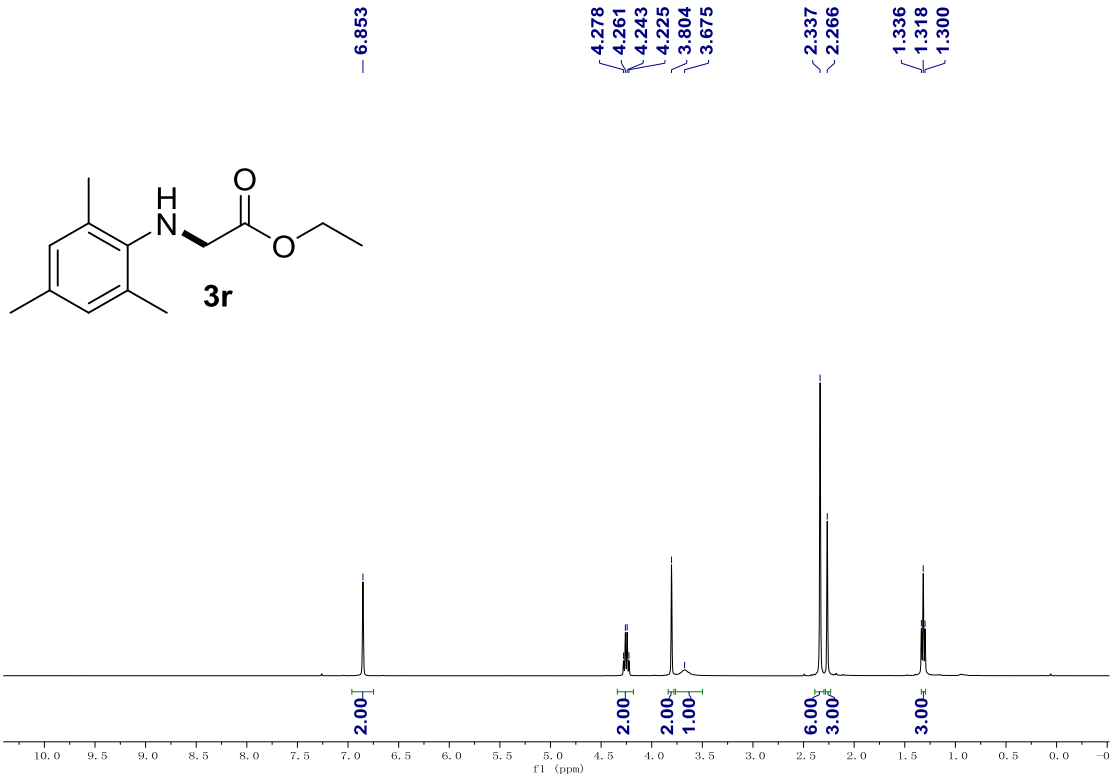
2.00
2.00
3.00
3.00
f1 (ppm)


171.18
147.05
139.07
129.14
119.11
113.82
110.10
61.24
45.88
21.56
14.16
3q
f1 (ppm)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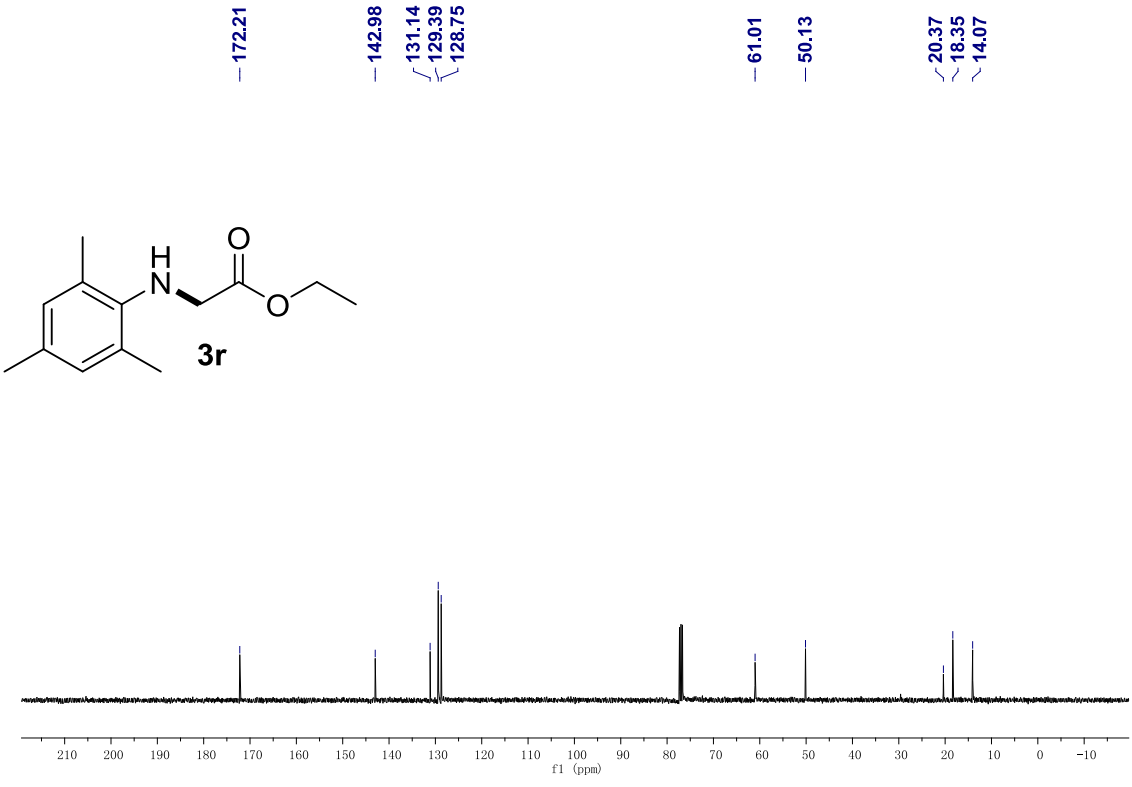
4.278
4.261
4.243
4.225
3.804
3.675
2.337
2.266
1.336
1.318
1.300
3r
2.00
2.00
2.00
1.00
6.00
3.00
3.00
f1 (ppm)


172.21
142.98
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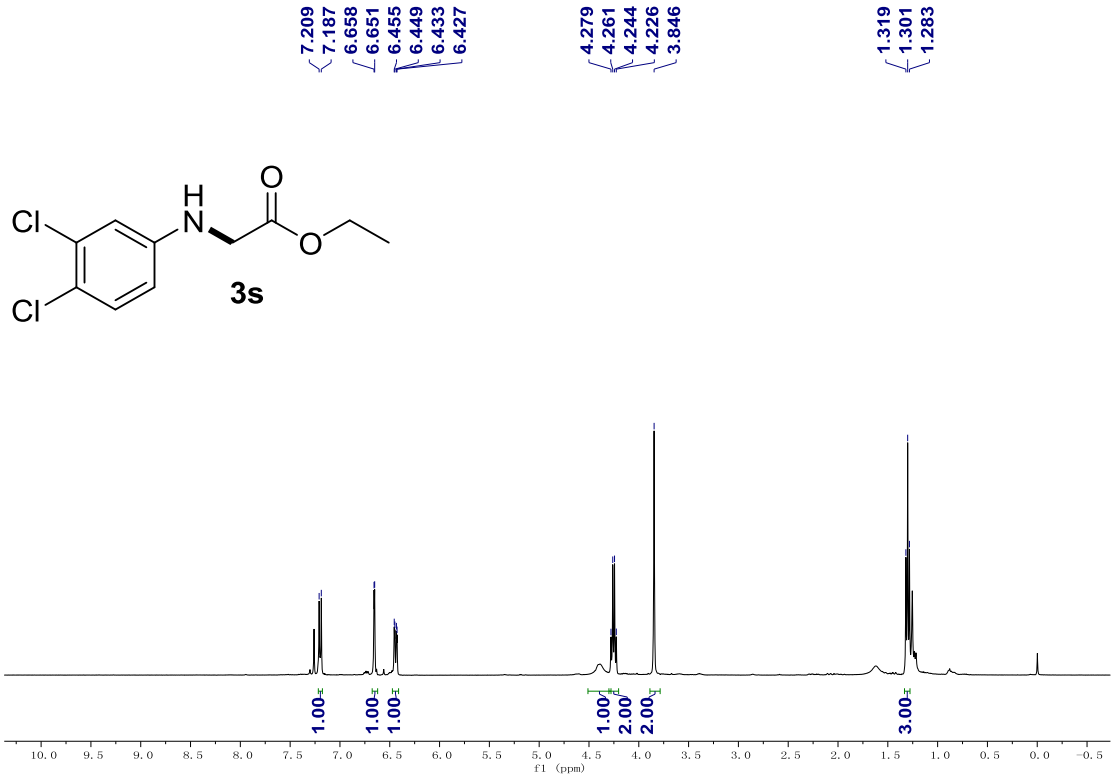
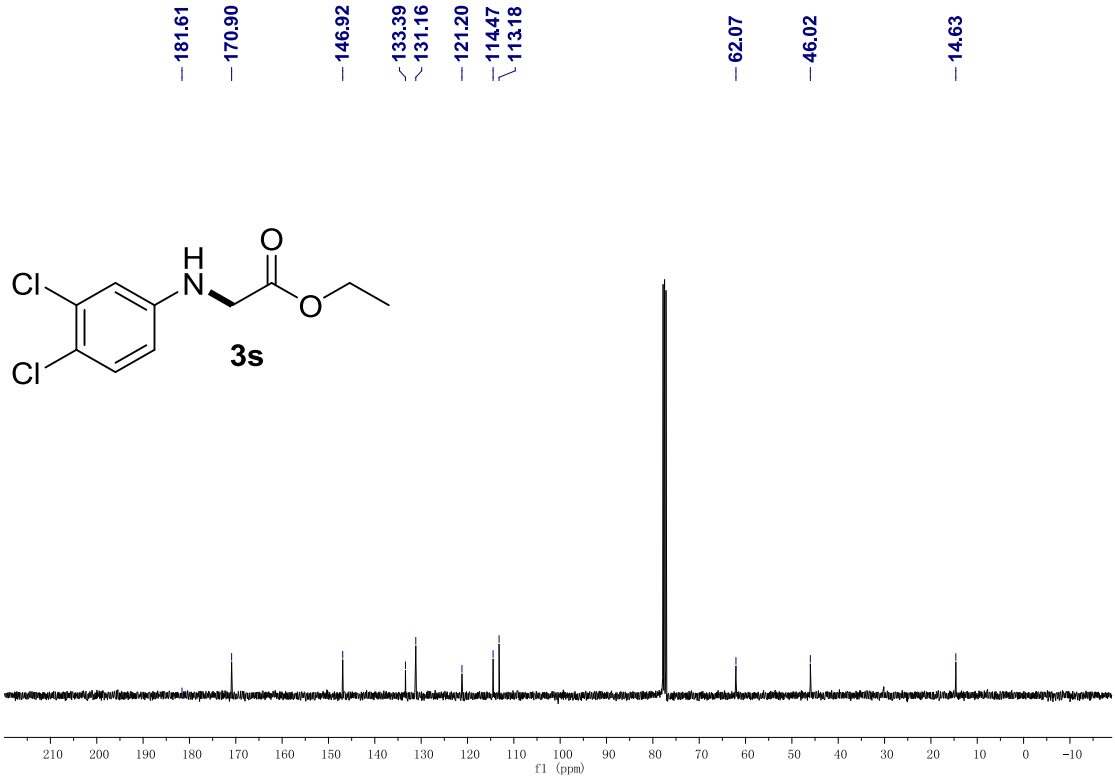
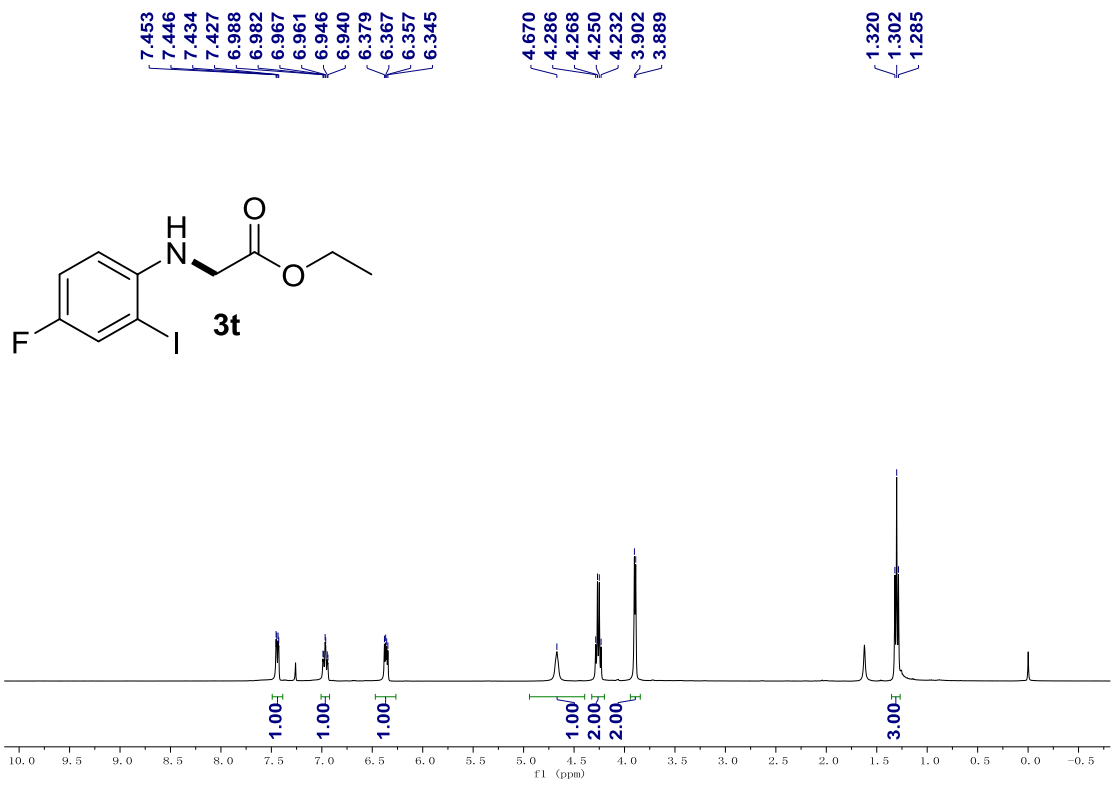
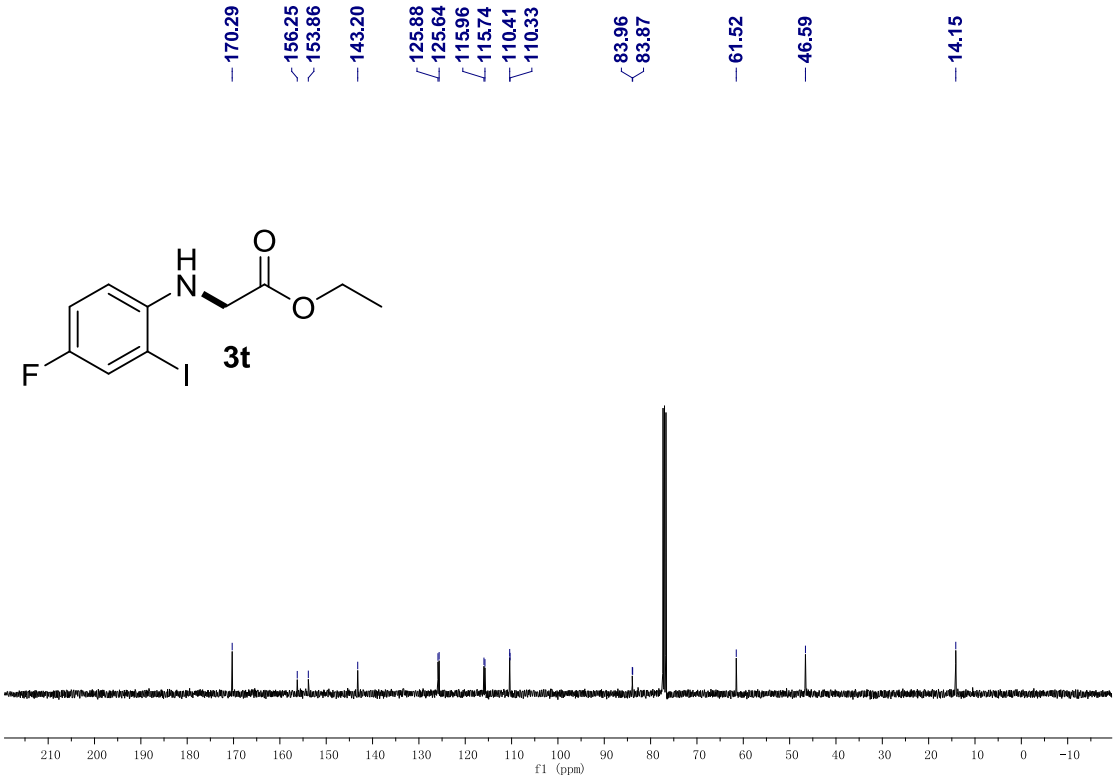
129.39
128.75
61.01
50.13
20.37
18.35
14.07
3r
f1 (ppm)


7.209
7.187
6.658
6.651
6.455
6.449
6.433
6.427
4.279
4.261
4.244
4.226
3.846
1.319
1.301
1.283
Cl
Cl
H
N
O
O
3s
1.00
1.00
1.00
1.00
2.00
2.00
3.00
f1 (ppm)

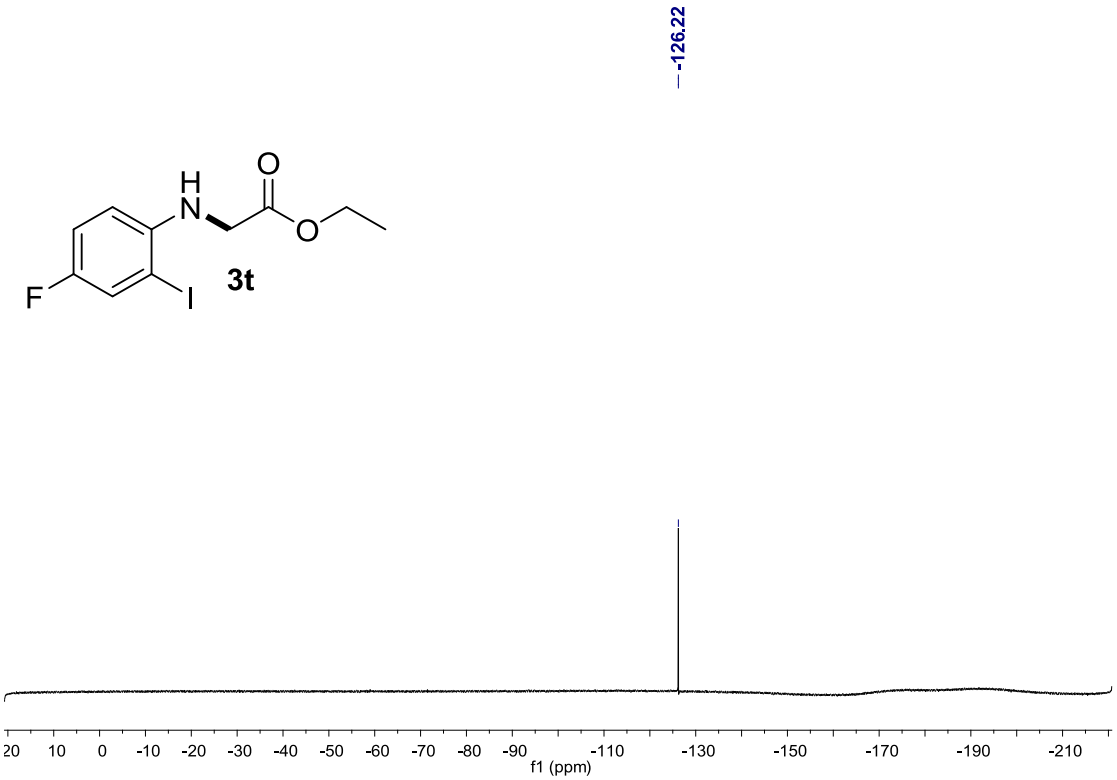

181.61
170.90
146.92
133.39
131.16
121.20
114.47
113.18
62.07
46.02
14.63
Cl
Cl
H
N
O
O
3s
f1 (ppm)

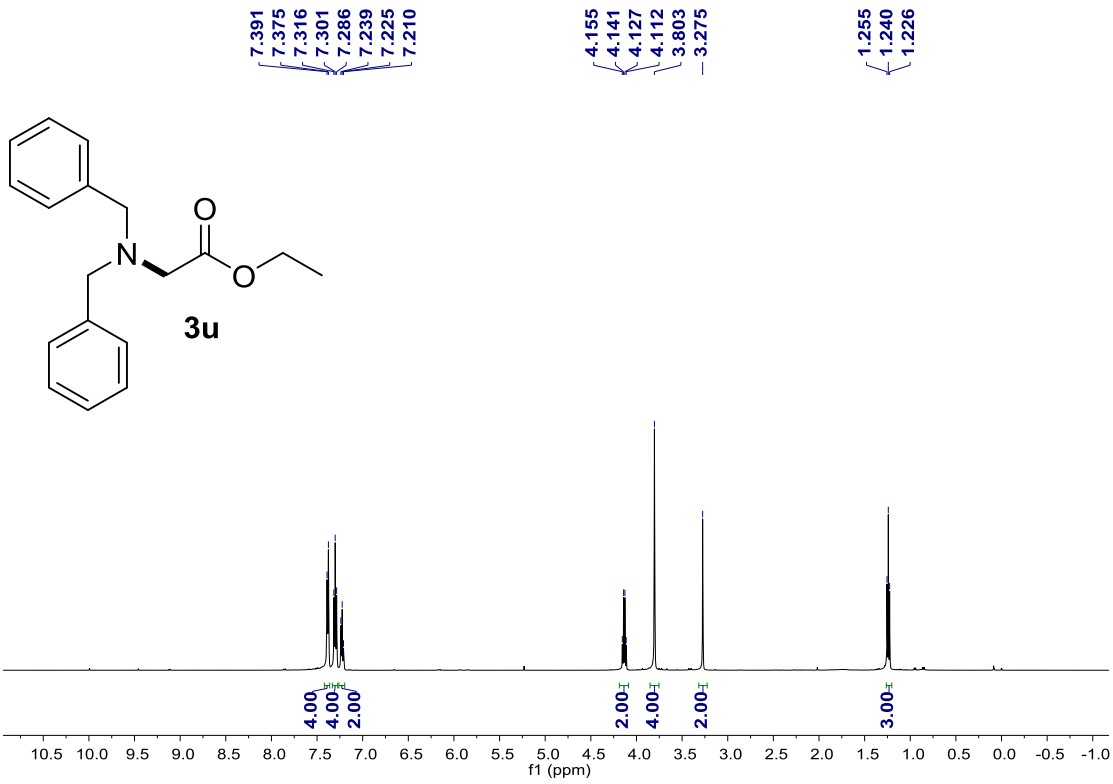

7.453
7.446
7.434
7.427
6.988
6.982
6.967
6.961
6.946
6.940
6.379
6.367
6.357
6.345
4.670
4.286
4.268
4.250
4.232
3.902
3.889
1.320
1.302
1.285
3t
1.00
1.00
1.00
1.00
2.00
2.00
3.00
f1 (ppm)

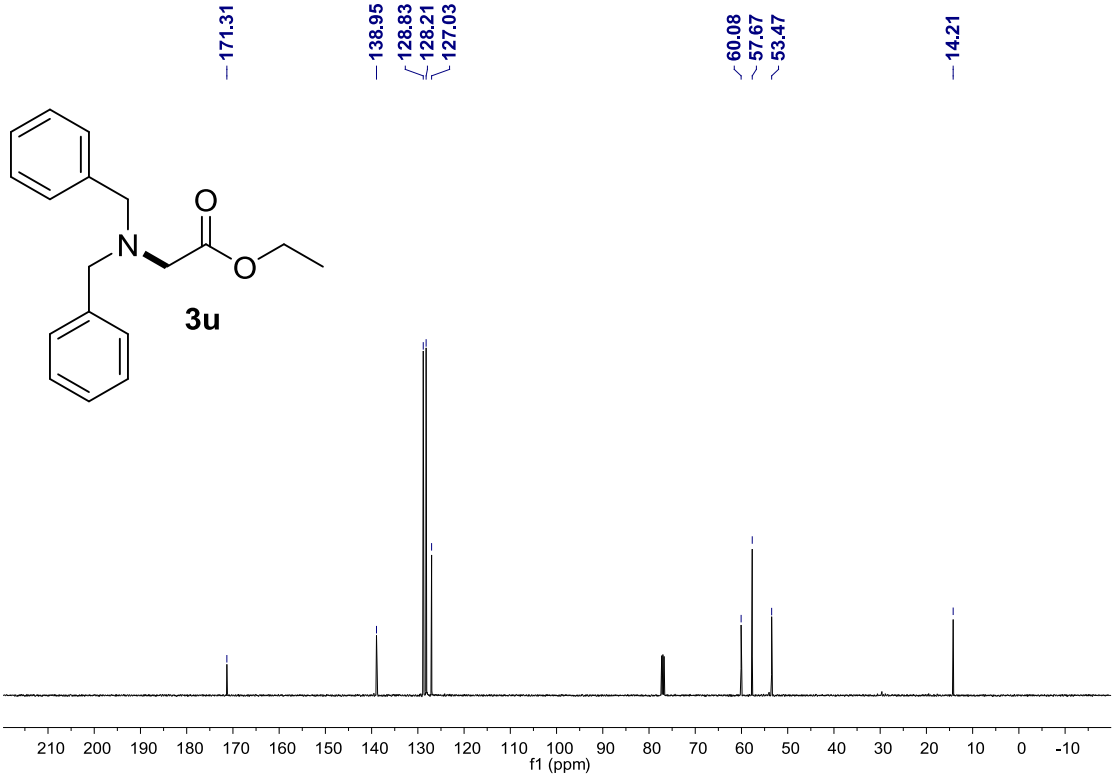

170.29
156.25
153.86
143.20
125.88
125.64
115.96
115.74
110.41
110.33
83.96
83.87
61.52
46.59
14.15
3t
f1 (ppm)

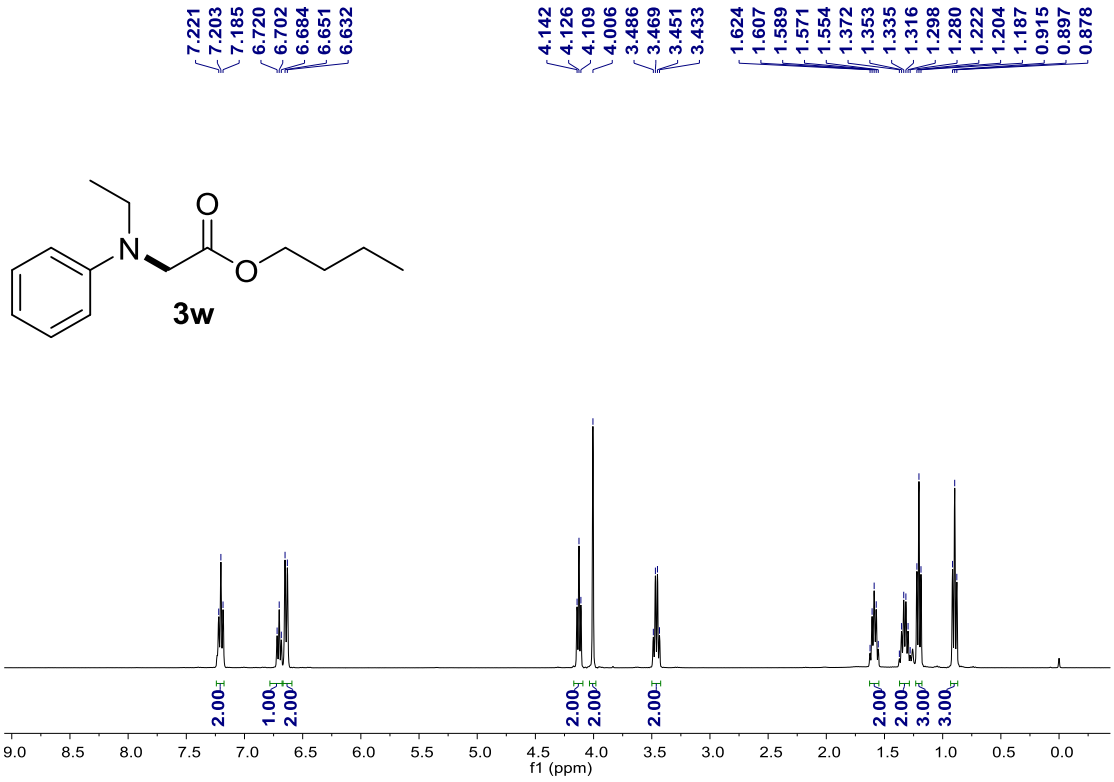
H
N
O
O
F
I

3t


-126.22
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)


7.391
7.375
7.316
7.301
7.286
7.239
7.225
7.210
4.155
4.141
4.127
4.112
3.803
3.275
1.255
1.240
1.226
O
N
O
3u
4.00
4.00
2.00
2.00
4.00
2.00
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


171.31
138.95
128.83
128.21
127.03
60.08
57.67
53.47
14.21
O
N
O
3u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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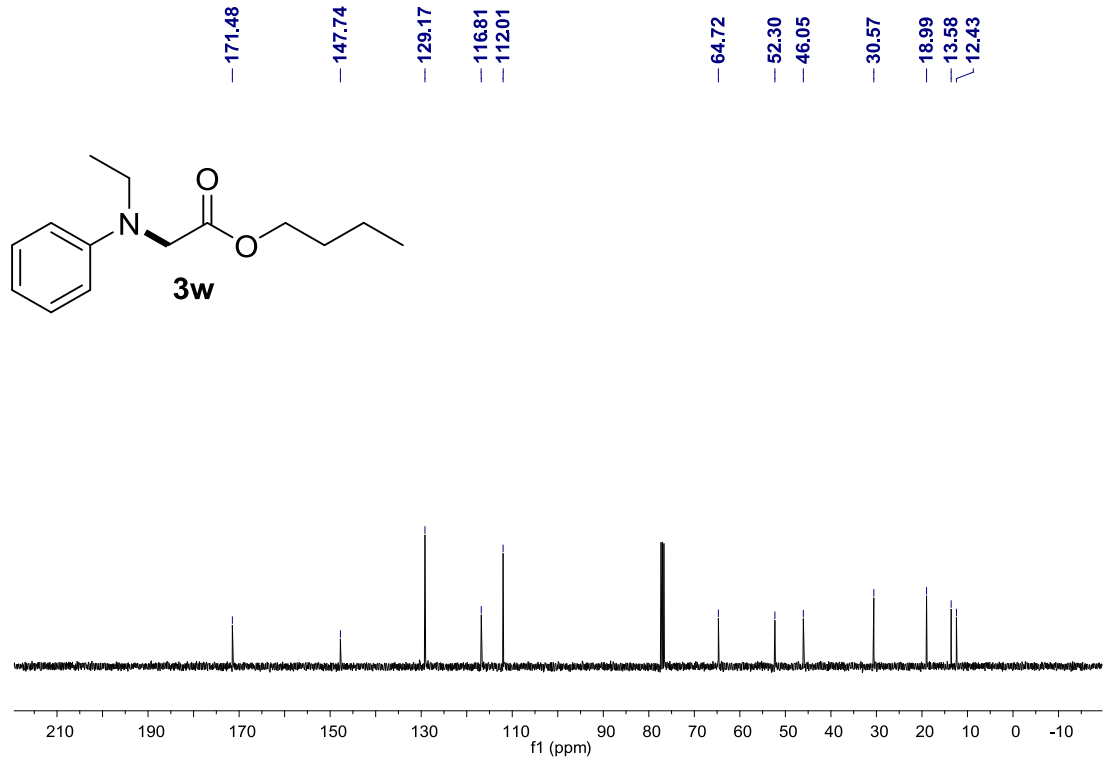
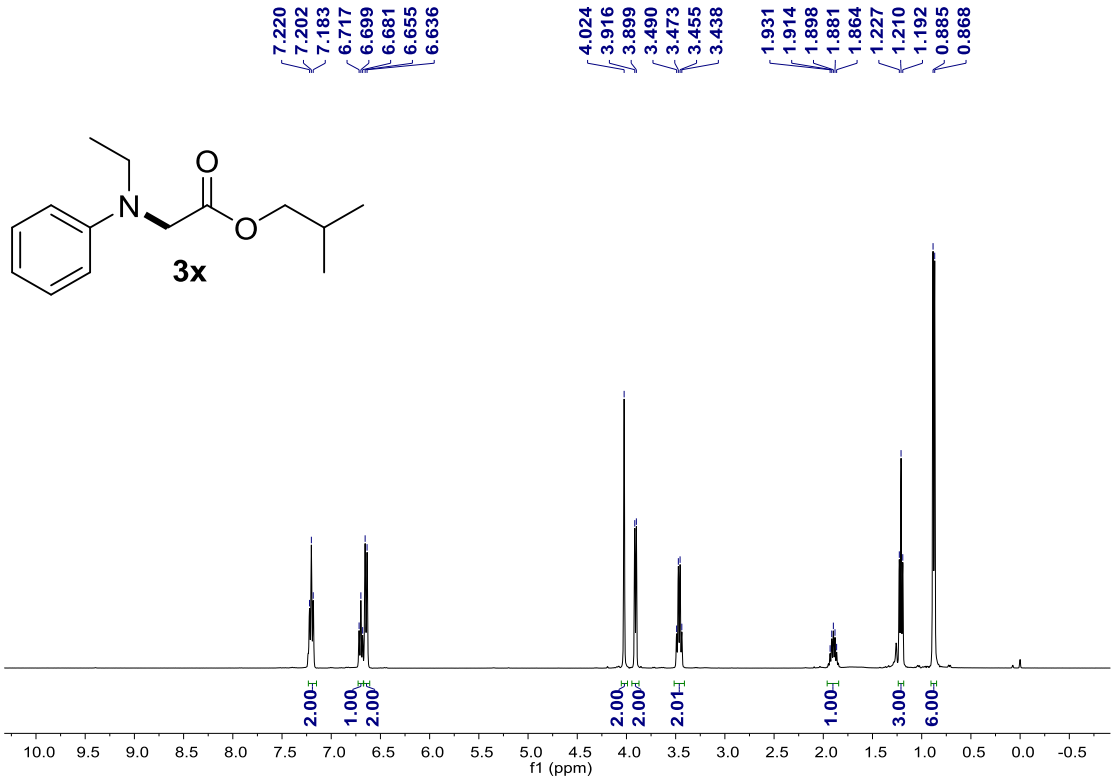
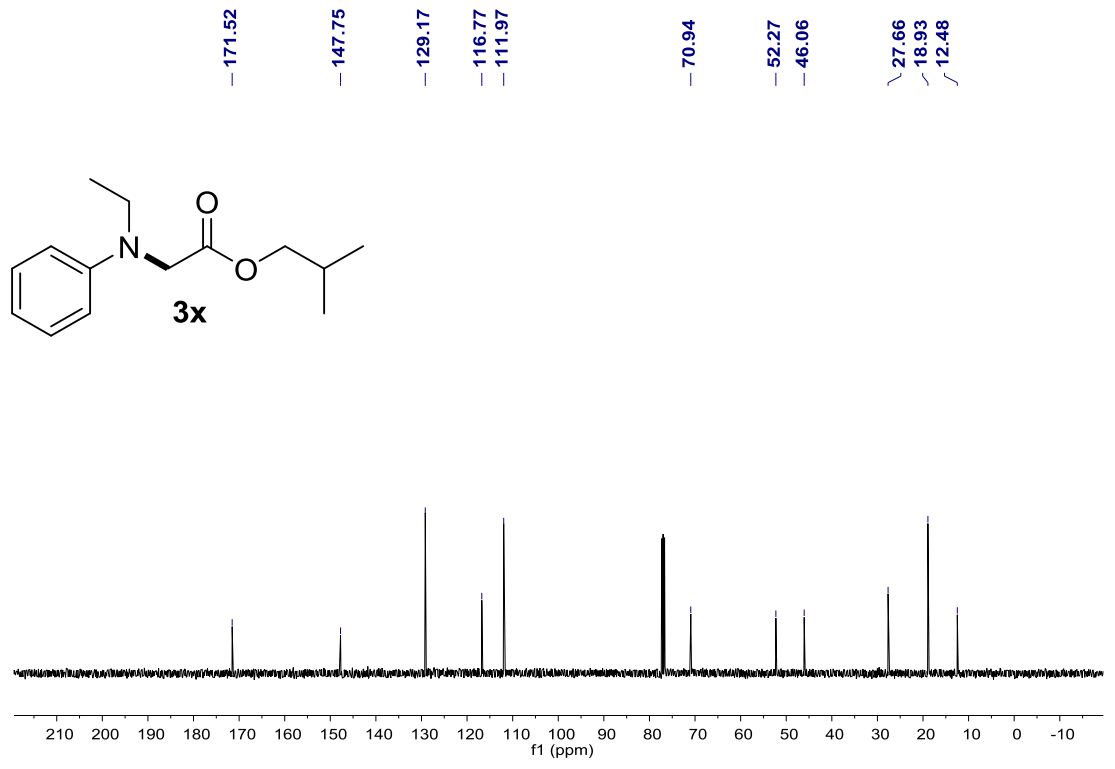
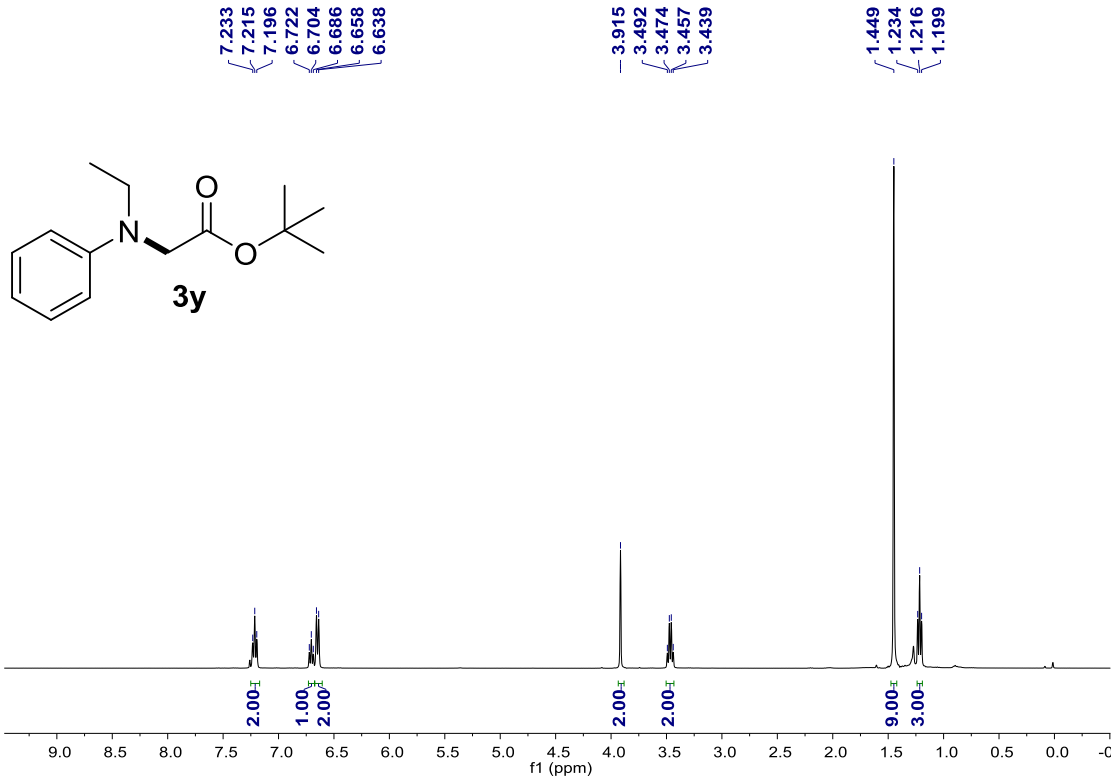
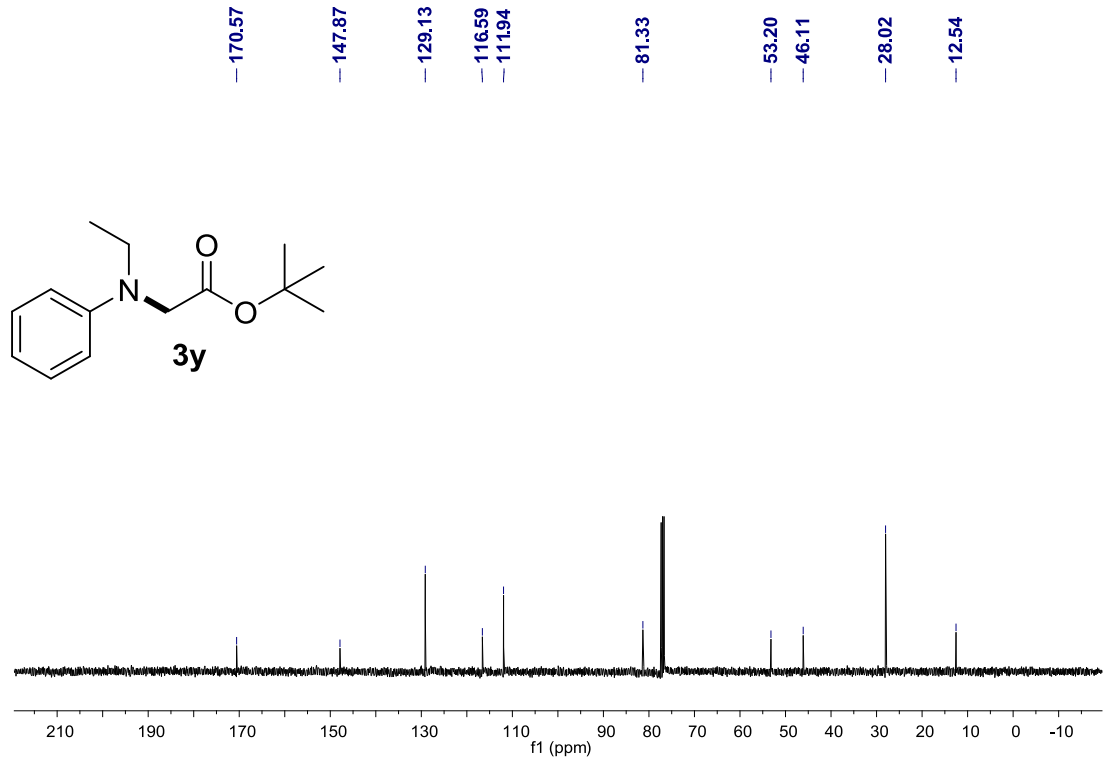
7.203
7.185
6.720
6.702
6.684
6.651
6.632
4.142
4.126
4.109
4.006
3.486
3.469
3.451
3.433
1.624
1.607
1.589
1.571
1.554
1.372
1.353
1.335
1.316
1.298
1.280
1.222
1.204
1.187
0.915
0.897
0.878
3w
2.00
1.00
2.00
2.00
2.00
2.00
2.00
2.00
3.00
3.00
f1 (ppm)

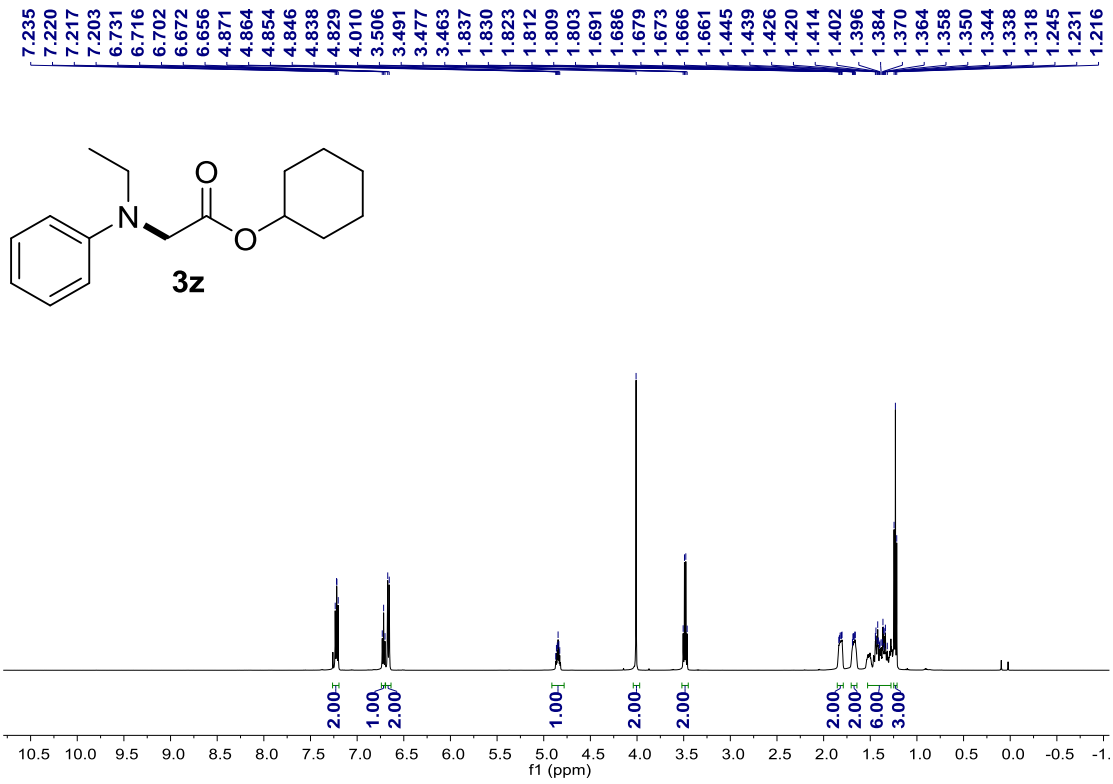

171.48
147.74
129.17
116.81
112.01
64.72
52.30
46.05
30.57
18.99
13.58
12.43
3w
f1 (ppm)

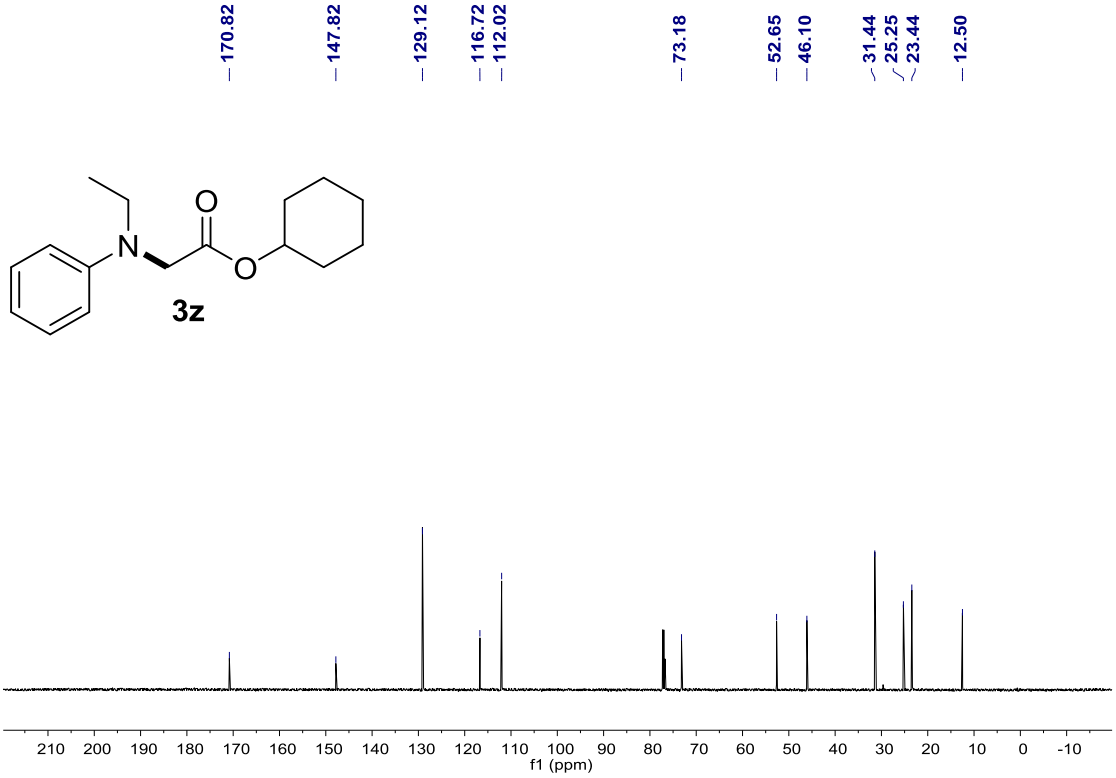

7.220
7.202
7.183
6.717
6.699
6.681
6.655
6.636
4.024
3.916
3.899
3.490
3.473
3.455
3.438
1.931
1.914
1.898
1.881
1.864
1.227
1.210
1.192
0.885
0.868
3x
2.00
1.00
2.00
2.00
2.00
2.01
1.00
3.00
6.00
f1 (ppm)

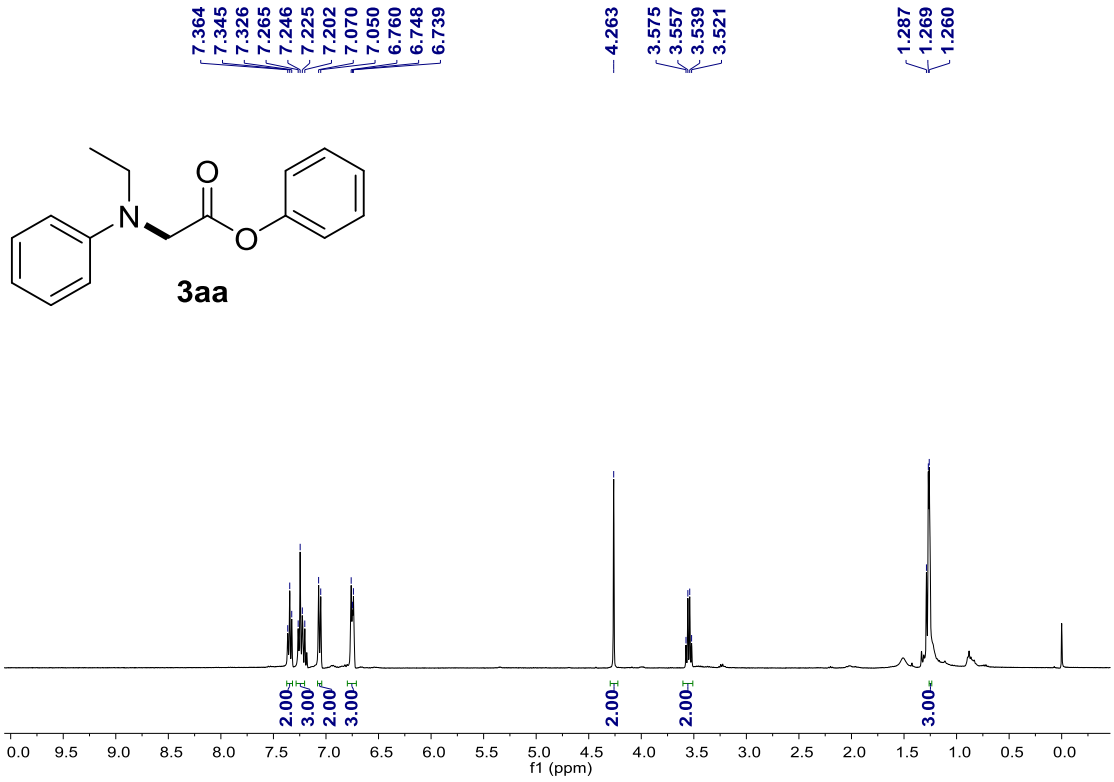

171.52
147.75
129.17
116.77
111.97
70.94
52.27
46.06
27.66
18.93
12.48
3x
f1 (ppm)

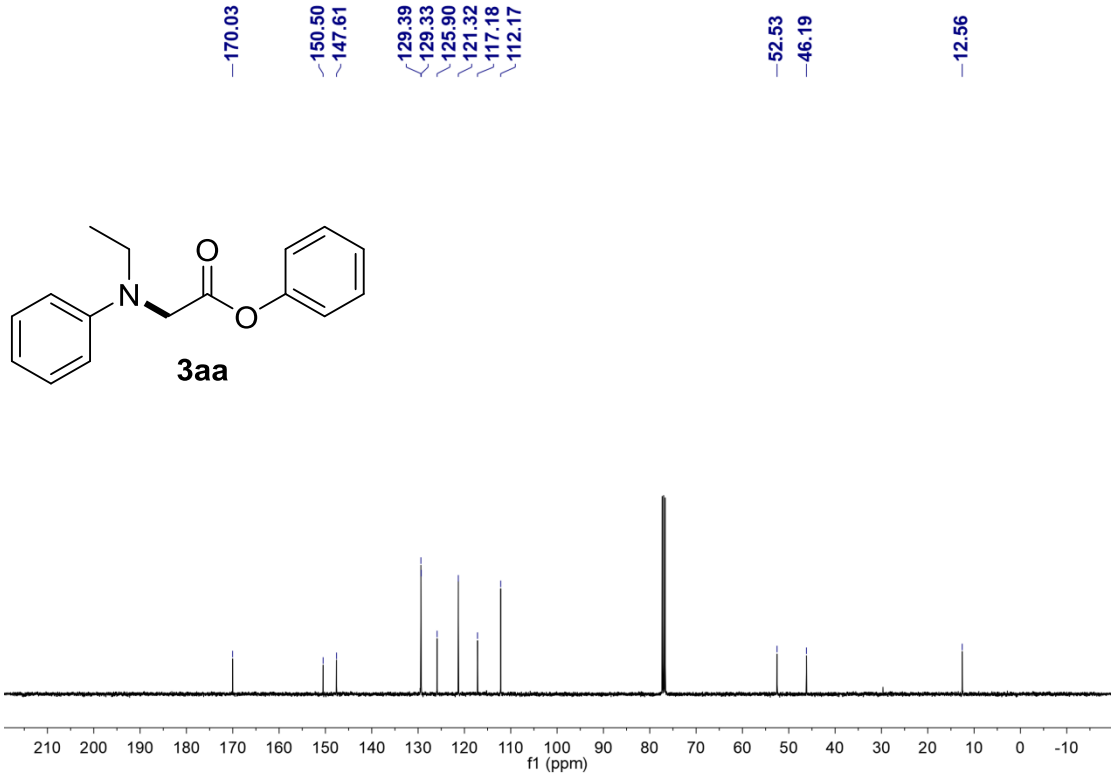

7.233
7.215
7.196
6.722
6.704
6.686
6.658
6.638
3.915
3.492
3.474
3.457
3.439
1.449
1.234
1.216
1.199
O
N
O
3y
2.00
1.00
2.00
2.00
2.00
9.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

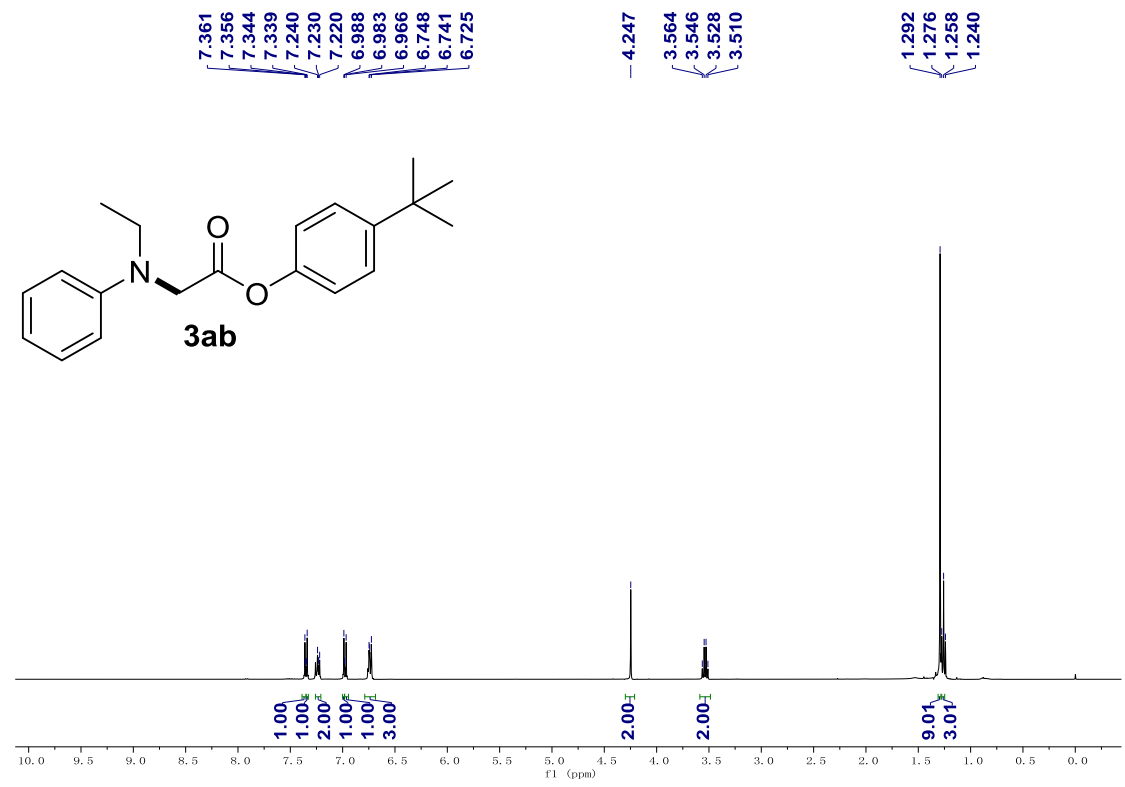

170.57
147.87
129.13
116.59
111.94
81.33
53.20
46.11
28.02
12.54
O
N
O
3y
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

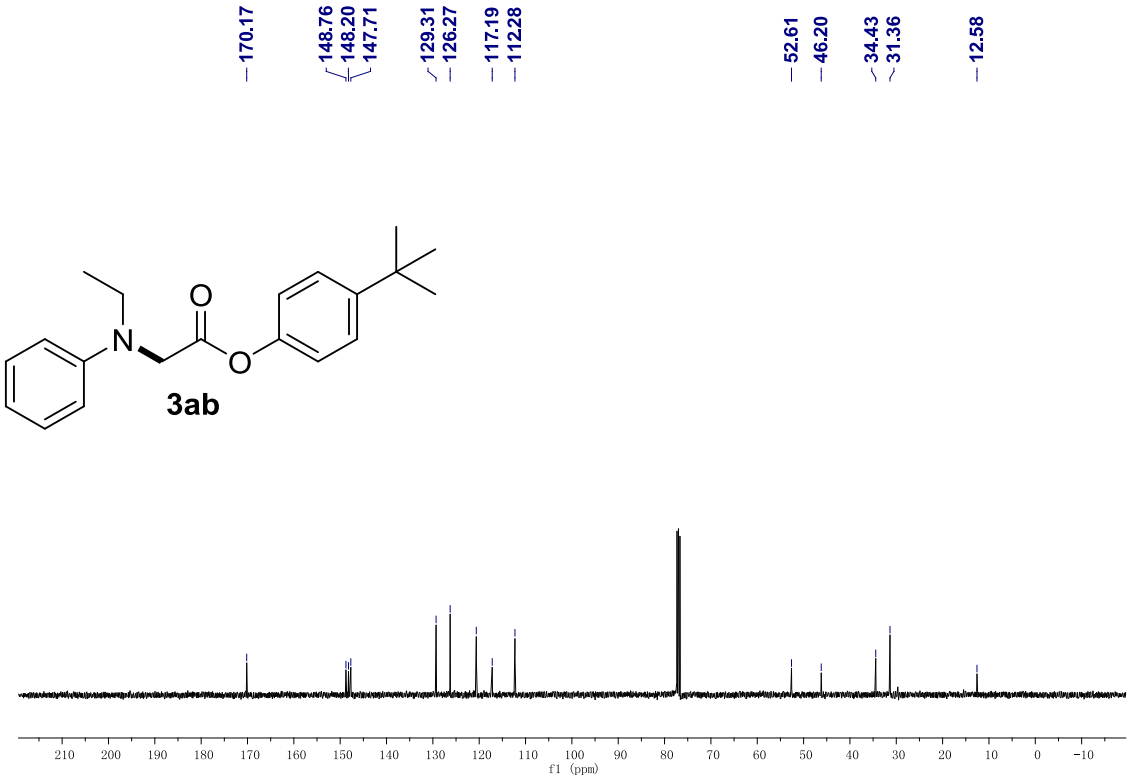

7.235
7.220
7.217
7.203
6.731
6.716
6.702
6.672
6.656
4.871
4.864
4.854
4.846
4.838
4.829
4.010
3.506
3.491
3.477
3.463
1.837
1.830
1.823
1.812
1.809
1.803
1.691
1.686
1.679
1.673
1.666
1.661
1.445
1.439
1.426
1.420
1.414
1.402
1.396
1.384
1.370
1.364
1.358
1.350
1.344
1.338
1.318
1.245
1.231
1.216
O
N
O
3z
2.00
1.00
2.00
1.00
2.00
2.00
2.00
2.00
6.00
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)

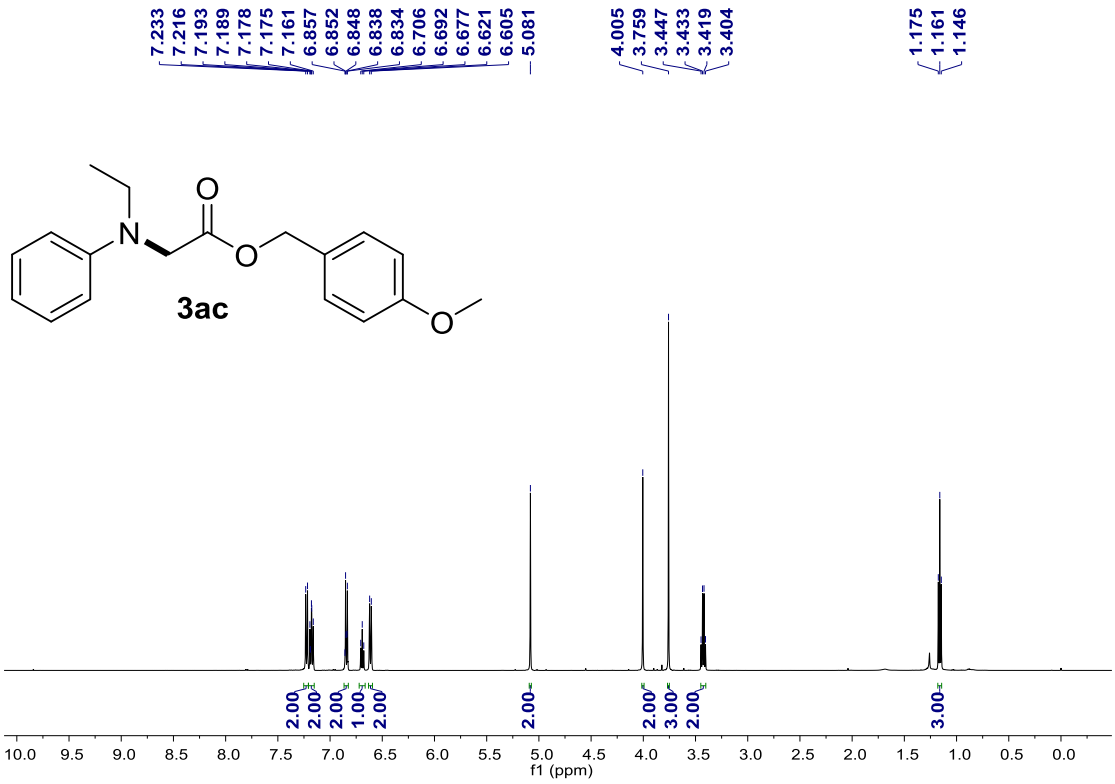

170.82
147.82
129.12
116.72
112.02
73.18
52.65
46.10
31.44
25.25
23.44
12.50
O
N
O
3z
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

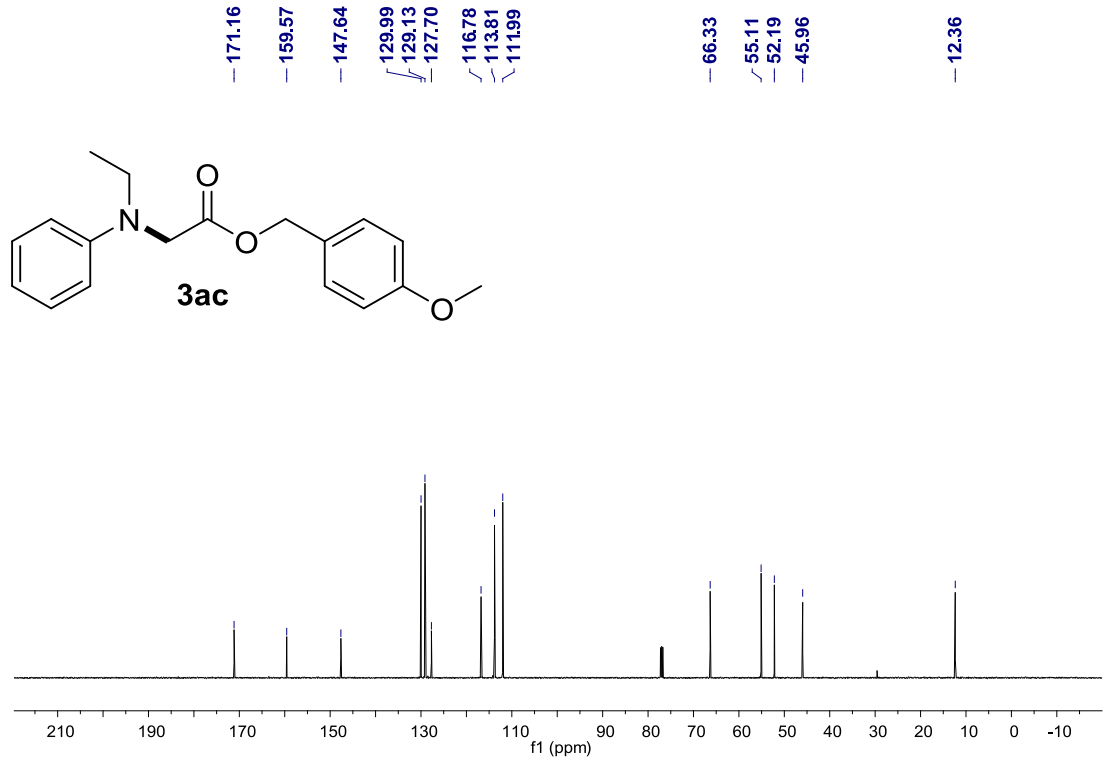

7.364
7.345
7.326
7.265
7.246
7.225
7.202
7.070
7.050
6.760
6.748
6.739
4.263
3.575
3.557
3.539
3.521
1.287
1.269
1.260
O
N
O
3aa
2.00
3.00
2.00
3.00
2.00
2.00
3.00
f1 (ppm)


170.03
150.50
147.61
129.39
129.33
125.90
121.32
117.18
112.17
52.53
46.19
12.56
O
N
O
3aa
f1 (ppm)


7.361
7.356
7.344
7.339
7.240
7.230
7.220
6.988
6.983
6.966
6.748
6.741
6.725
4.247
3.564
3.546
3.528
3.510
1.292
1.276
1.258
1.240
O
N
O
3ab
1.00
1.00
2.00
1.00
1.00
3.00
2.00
2.00
9.01
3.01
f1 (ppm)


170.17
148.76
148.20
147.71
129.31
126.27
117.19
112.28
52.61
46.20
34.43
31.36
12.58
O
N
O
3ab
f1 (ppm)


7.233
7.216
7.193
7.189
7.178
7.175
7.161
6.857
6.852
6.848
6.838
6.834
6.706
6.692
6.677
6.621
6.605
5.081
4.005
3.759
3.447
3.433
3.419
3.404
1.175
1.161
1.146
3ac
2.00
2.00
2.00
1.00
2.00
2.00
2.00
3.00
2.00
3.00
f1 (ppm)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159.57
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127.70
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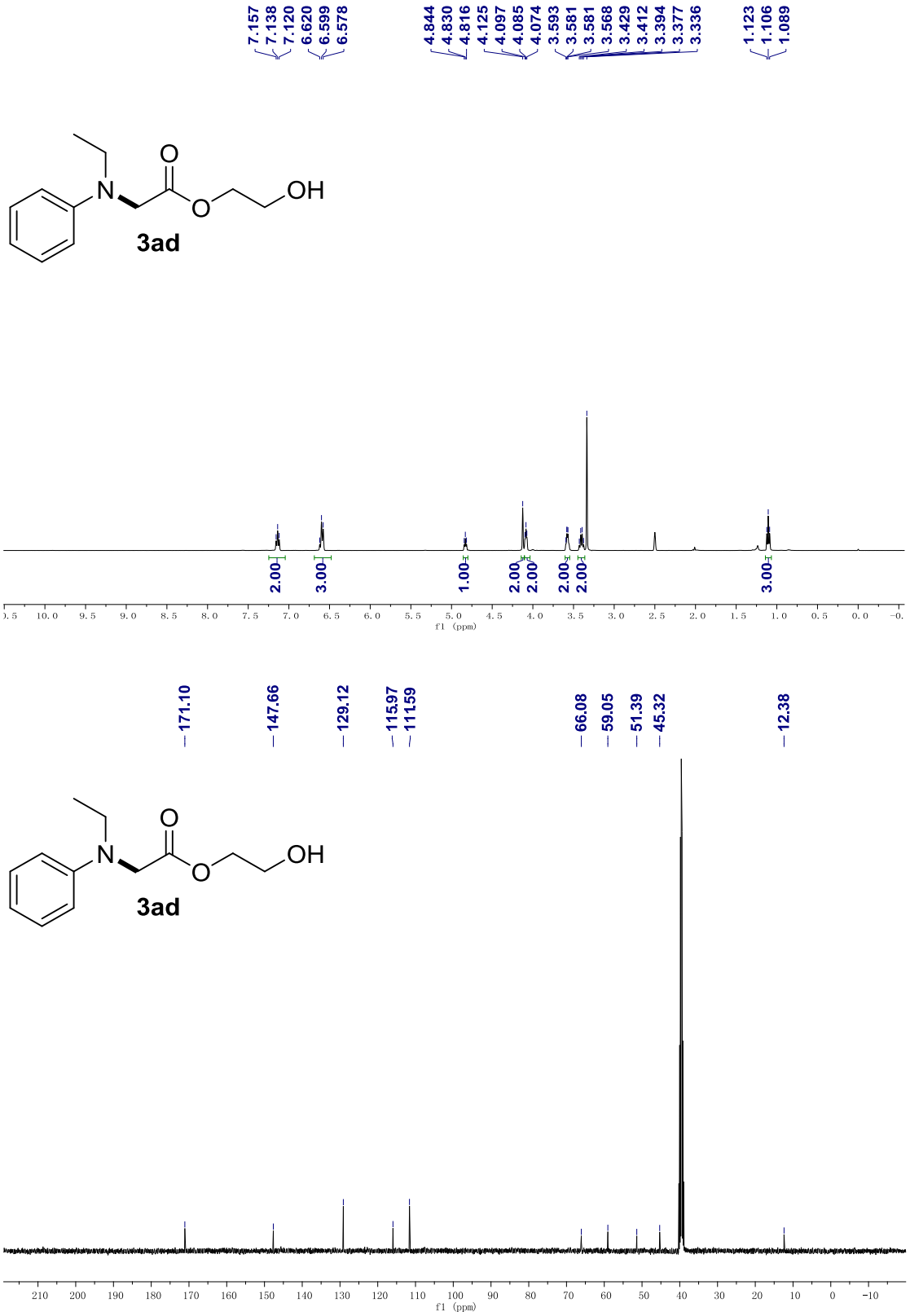
113.81
111.99
66.33
55.11
52.19
45.96
12.36
3ac
f1 (ppm)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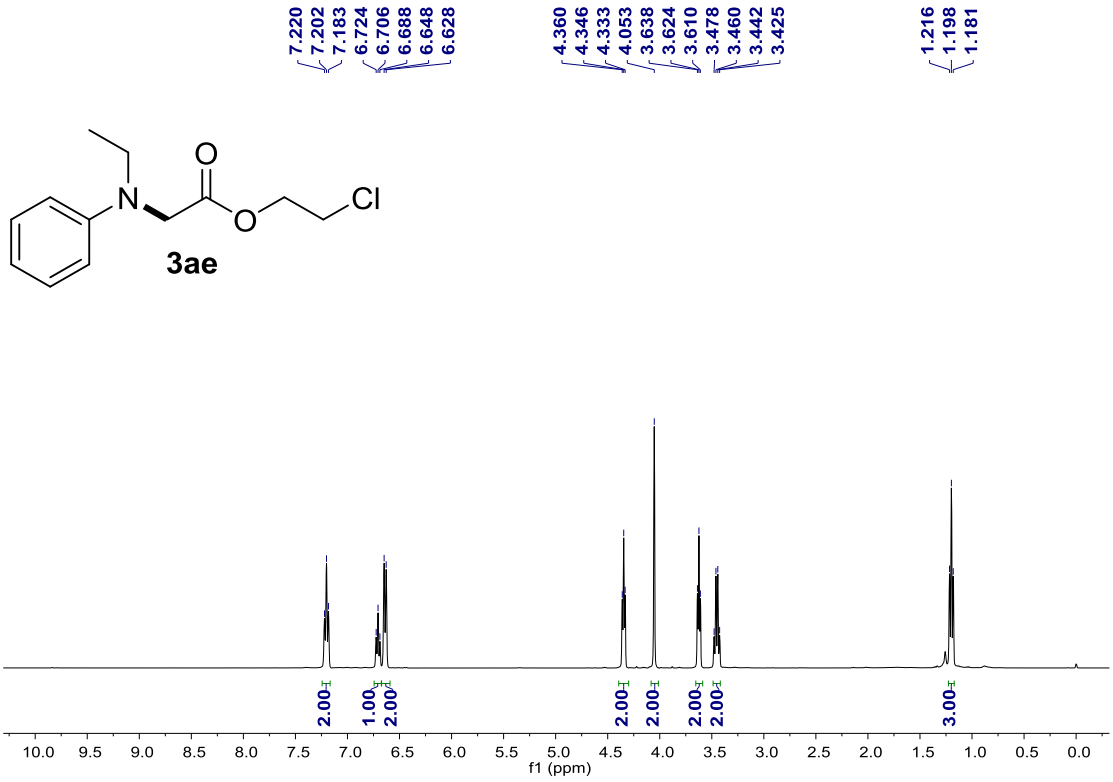
7.138
7.120
6.620
6.599
6.578
4.844
4.830
4.816
4.125
4.097
4.085
4.074
3.593
3.581
3.581
3.568
3.429
3.412
3.394
3.377
3.336
1.123
1.106
1.089
O
N
O
OH
3ad
2.00
3.00
1.00
2.00
2.00
2.00
2.00
3.00
f1 (ppm)
171.10
147.66
129.12
115.97
111.59
66.08
59.05
51.39
45.32
12.38
O
N
O
OH
3ad
f1 (ppm)


7.220
7.202
7.183
6.724
6.706
6.688
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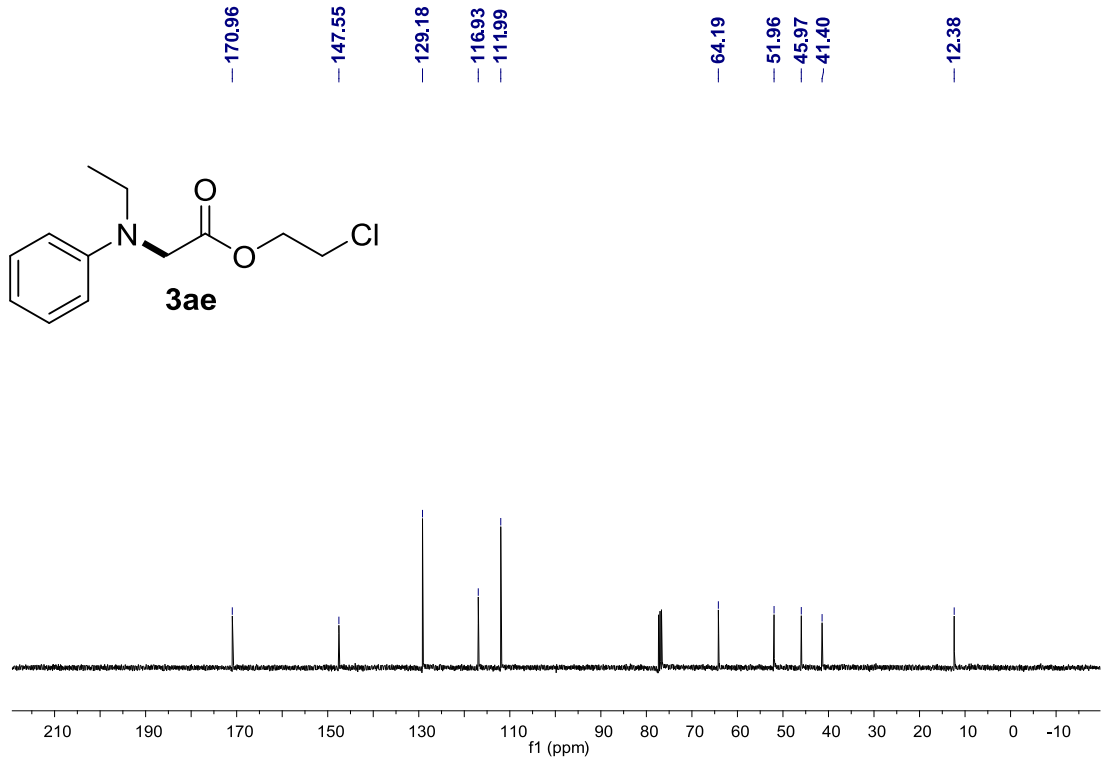
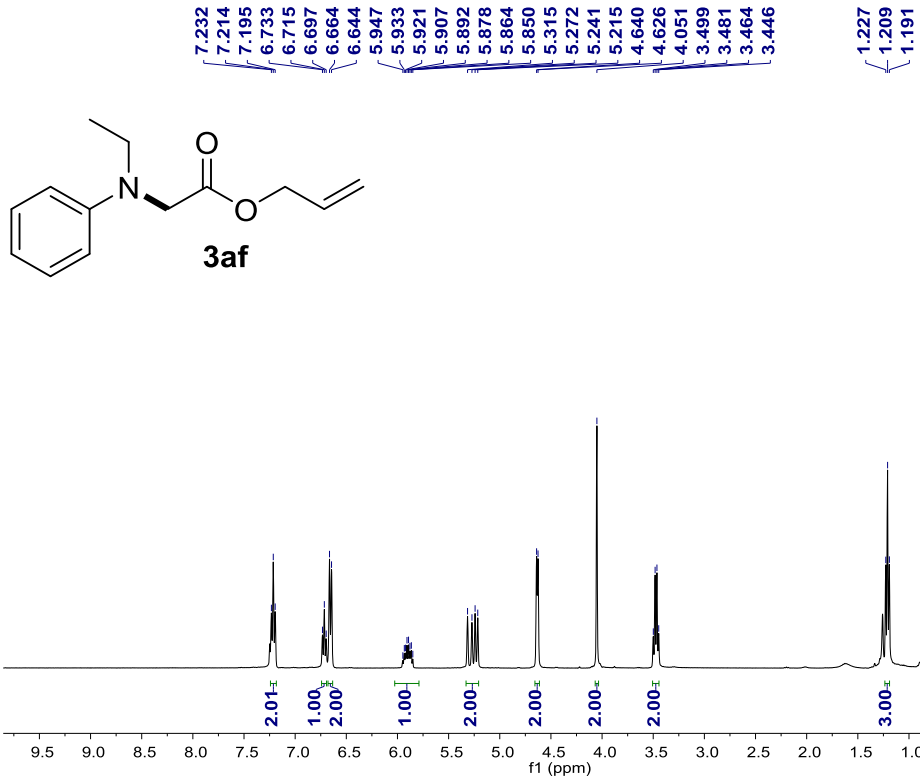
6.628
4.360
4.346
4.333
4.053
3.638
3.624
3.610
3.478
3.460
3.442
3.425
1.216
1.198
1.181
O
N
O
Cl
3ae
2.00
1.00
2.00
2.00
2.00
2.00
2.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


170.96
147.55
129.18
116.93
111.99
64.19
51.96
45.97
41.40
12.38
O
N
O
Cl
3ae
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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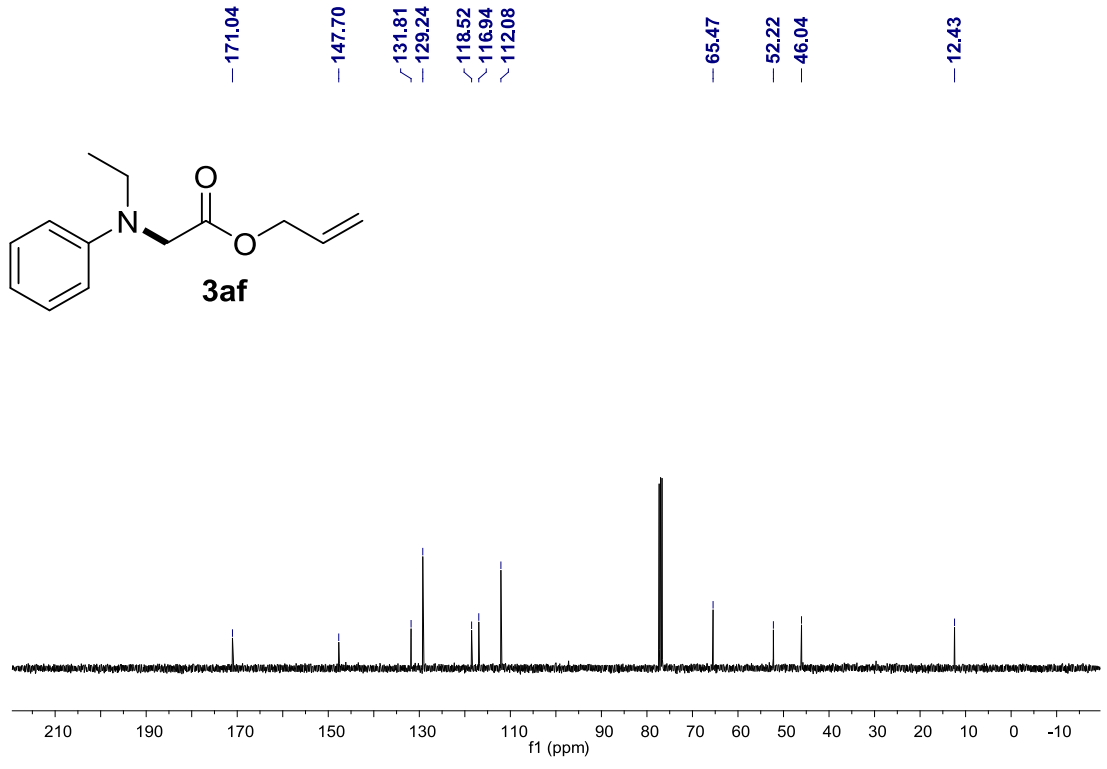
7.214
7.195
6.733
6.715
6.697
6.664
6.644
5.947
5.933
5.921
5.907
5.892
5.878
5.864
5.850
5.315
5.272
5.241
5.215
4.640
4.626
4.051
3.499
3.481
3.464
3.446
1.227
1.209
1.191
O
N
O
3af
2.01
1.00
2.00
1.00
2.00
2.00
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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118.52
116.94
112.08
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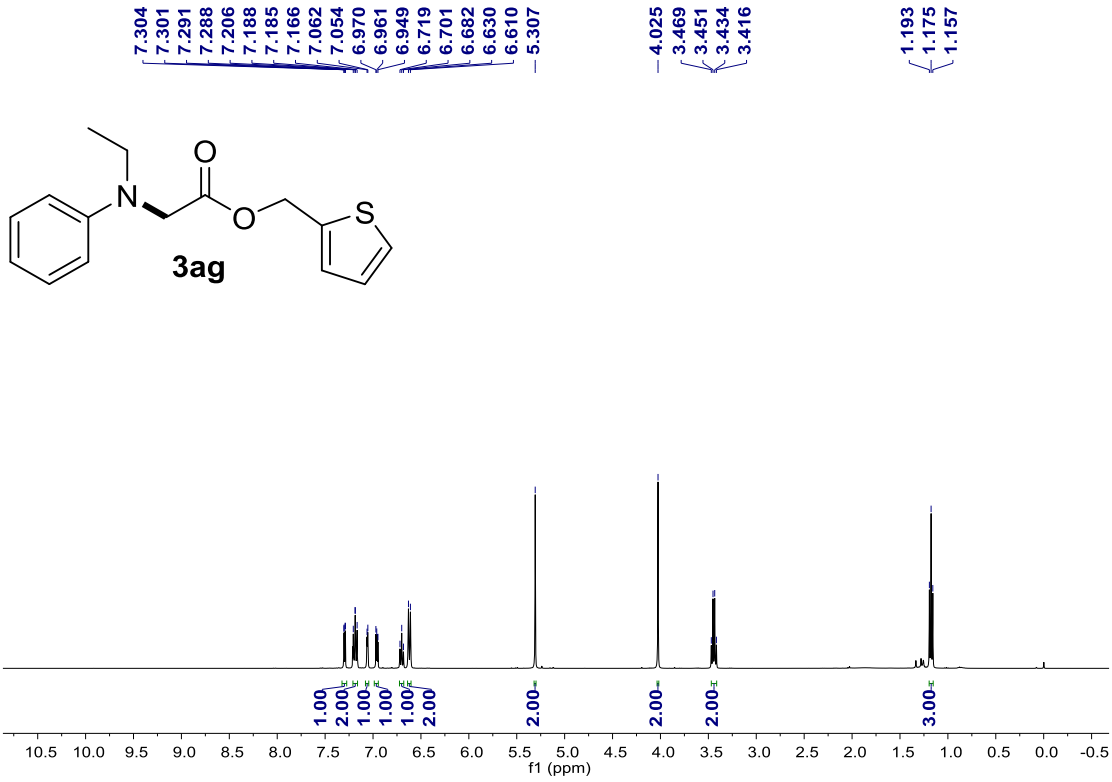
52.22
46.04
12.43
O
N
O
3af
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.288
7.206
7.188
7.185
7.166
7.062
7.054
6.970
6.961
6.949
6.719
6.701
6.682
6.630
6.610
5.307
4.025
3.469
3.451
3.434
3.416
1.193
1.175
1.157
3ag
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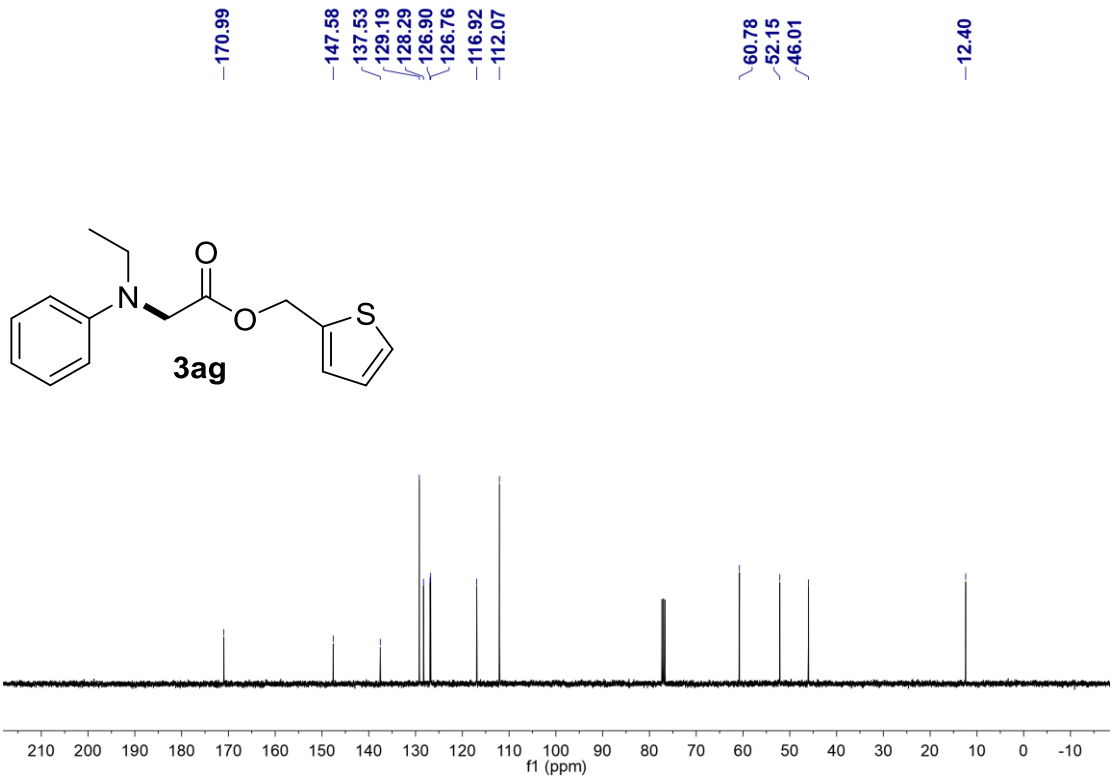
2.00
1.00
1.00
1.00
2.00
2.00
2.00
2.00
3.00
f1 (ppm)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147.58
137.53
129.19
128.29
126.90
126.76
116.92
112.07
60.78
52.15
46.01
12.40
3ag
f1 (ppm)


7.238
7.223
7.220
7.206
6.770
6.754
6.738
4.436
4.421
4.407
4.393
4.189
4.181
4.174
4.167
4.159
4.153
4.145
4.138
4.131
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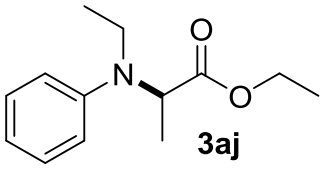
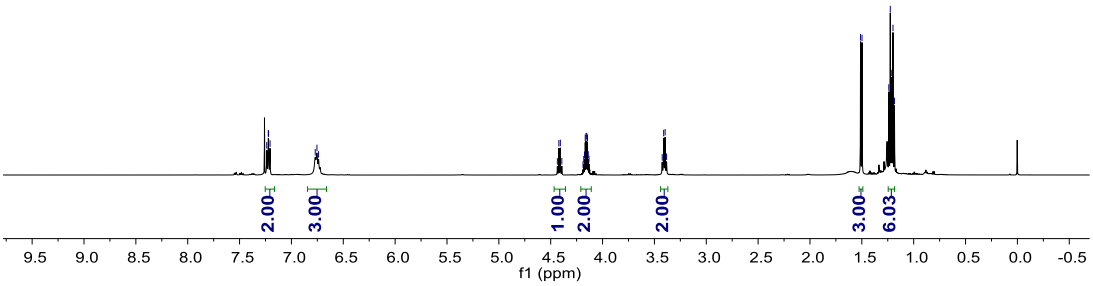
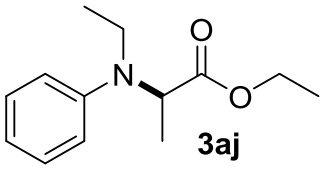
1.211
1.200
1.185


O
N
O

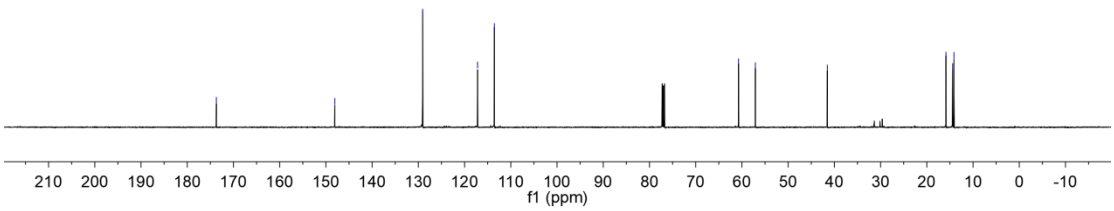
3aj

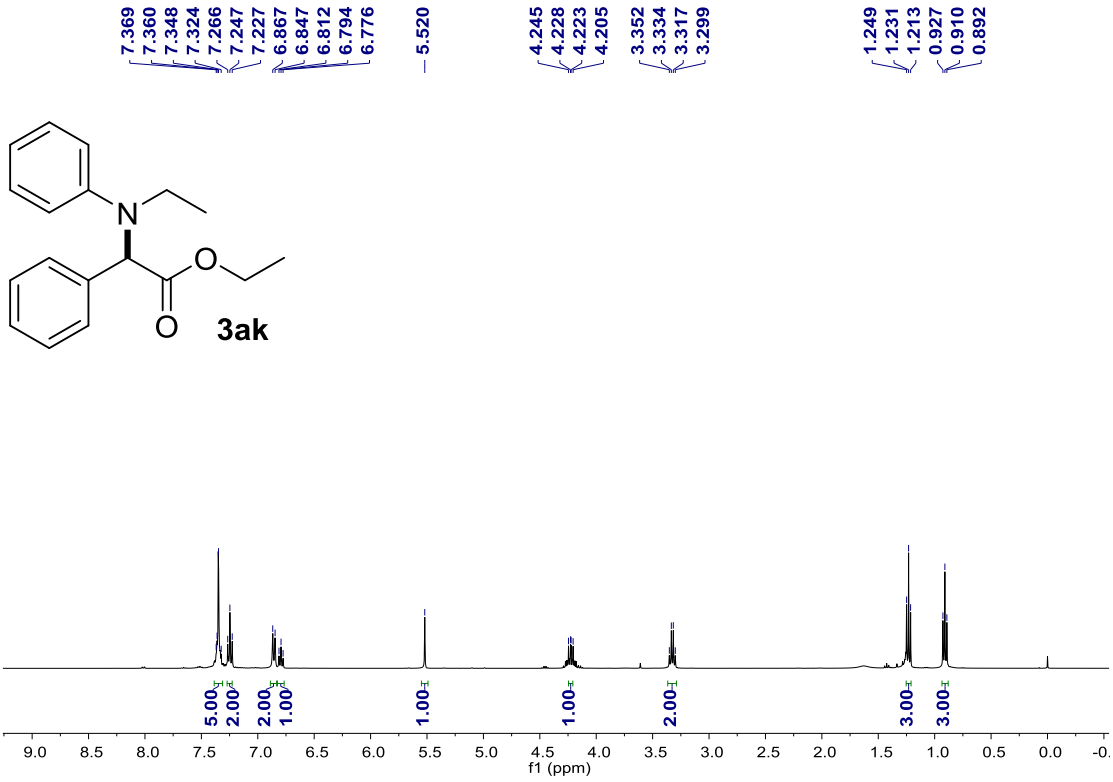

2.00
3.00
1.00
2.00
2.00
3.00
6.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


173.70
148.07
129.07
117.19
113.56
60.73
57.09
41.53
15.86
14.41
14.13


O
N
O

3aj


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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6.812
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4.245
4.228
4.223
4.205
3.352
3.334
3.317
3.299
1.249
1.231
1.213
0.927
0.910
0.892
3ak
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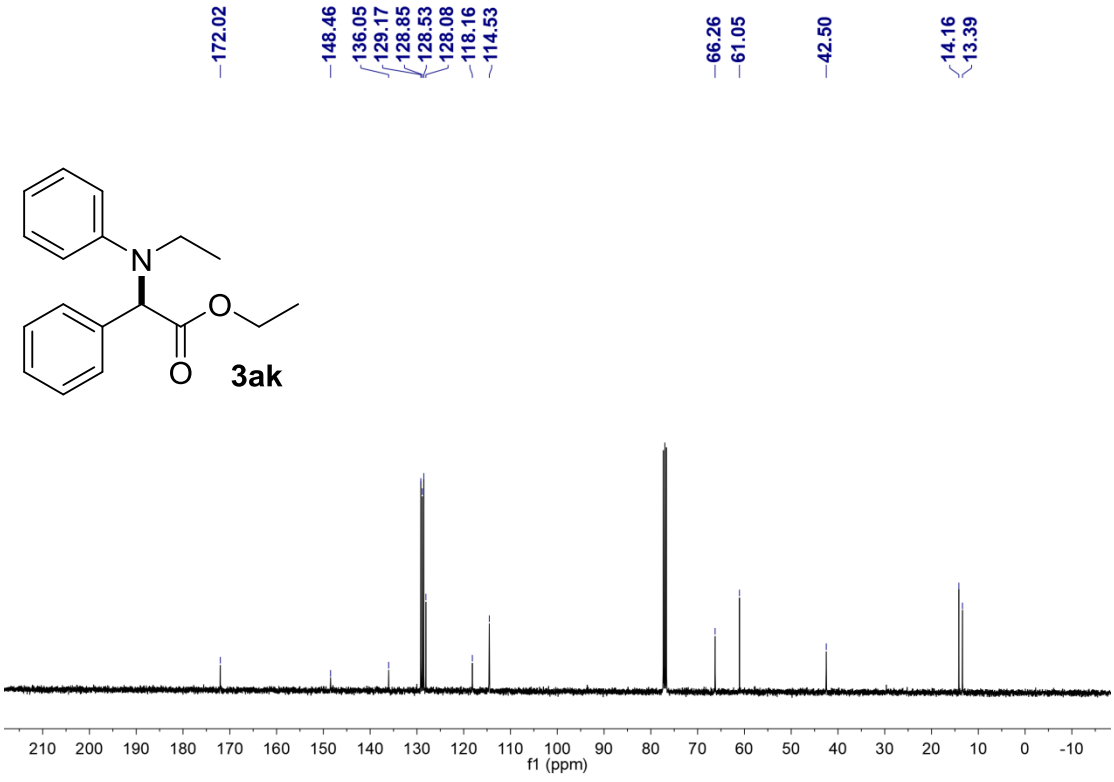
2.00
2.00
1.00
1.00
1.00
2.00
3.00
3.00
f1 (ppm)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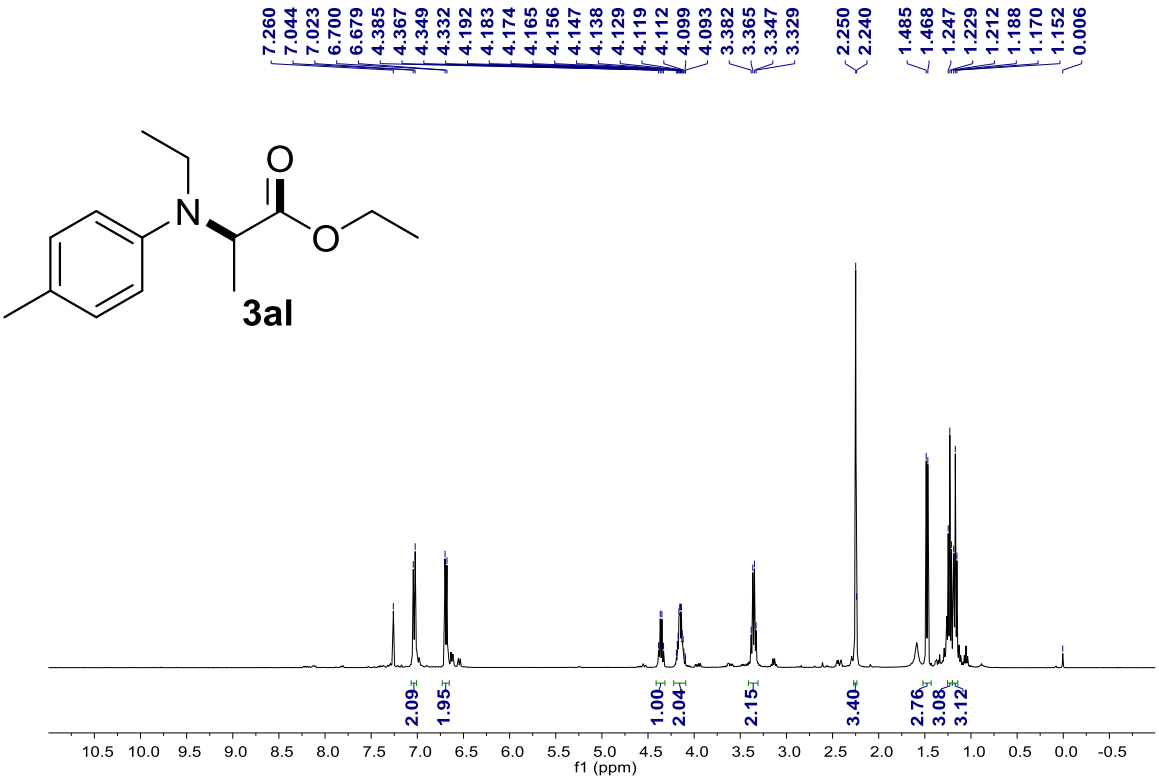
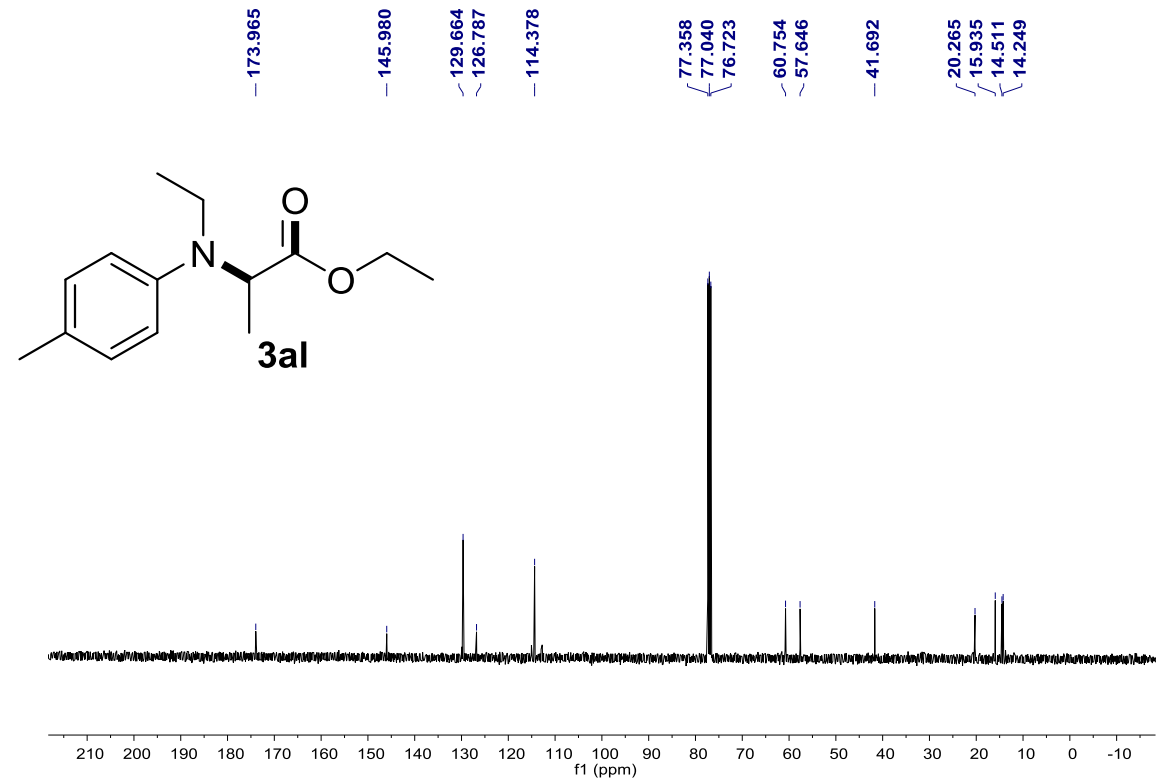
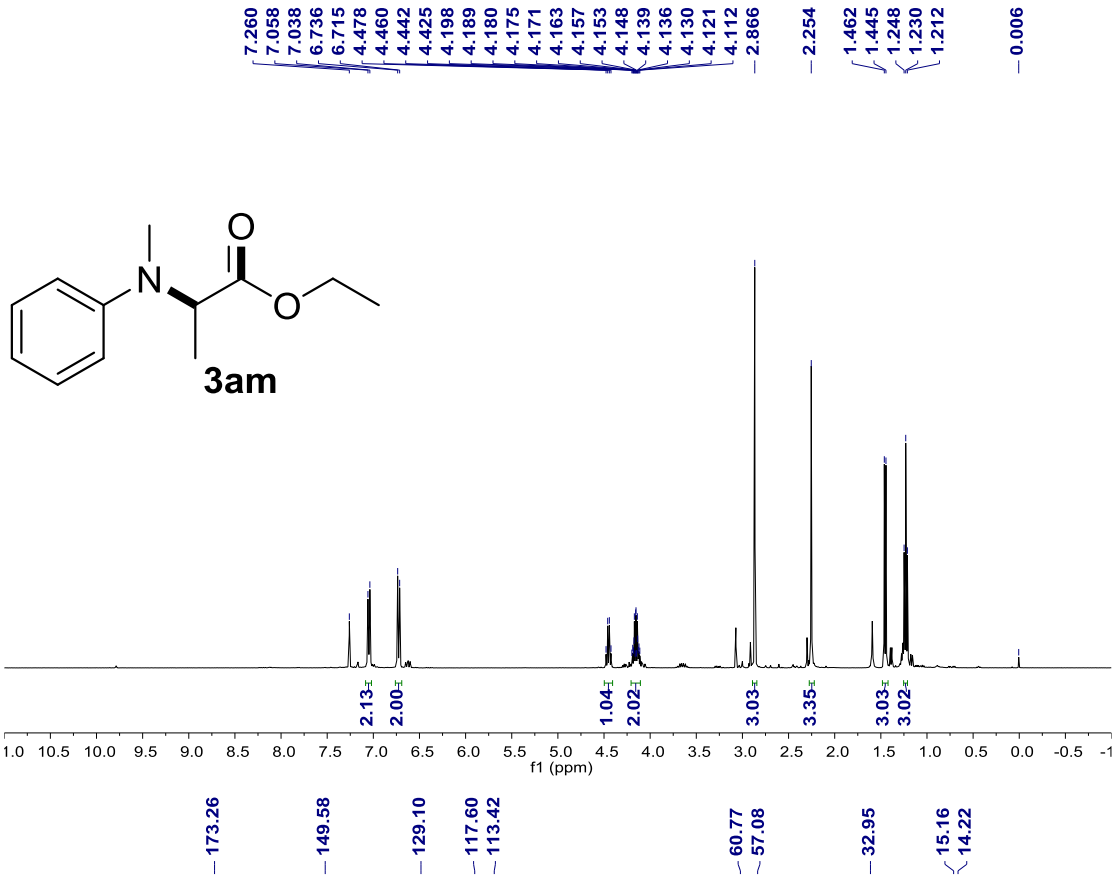
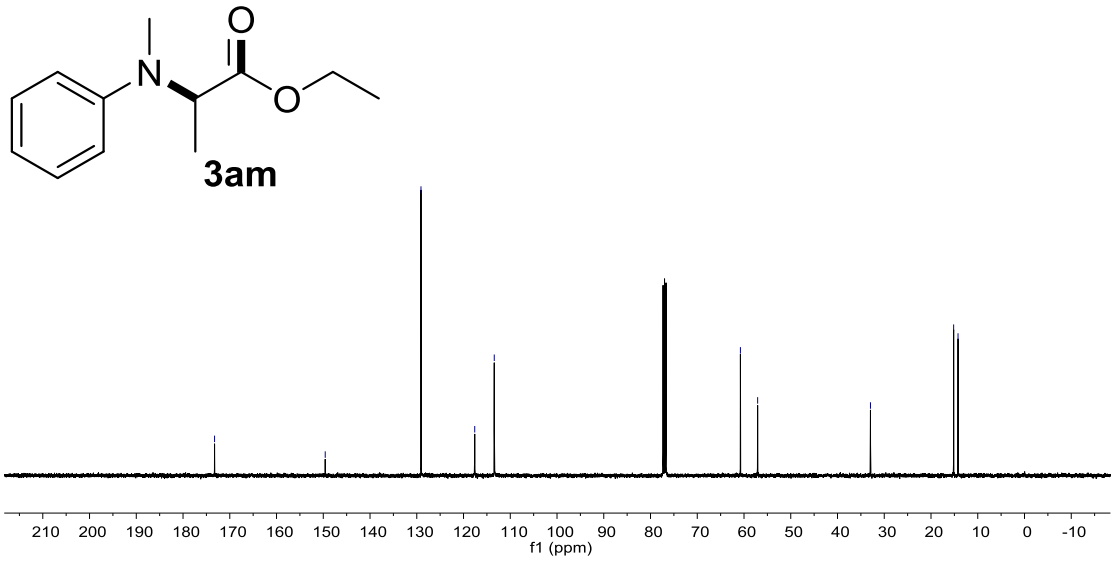
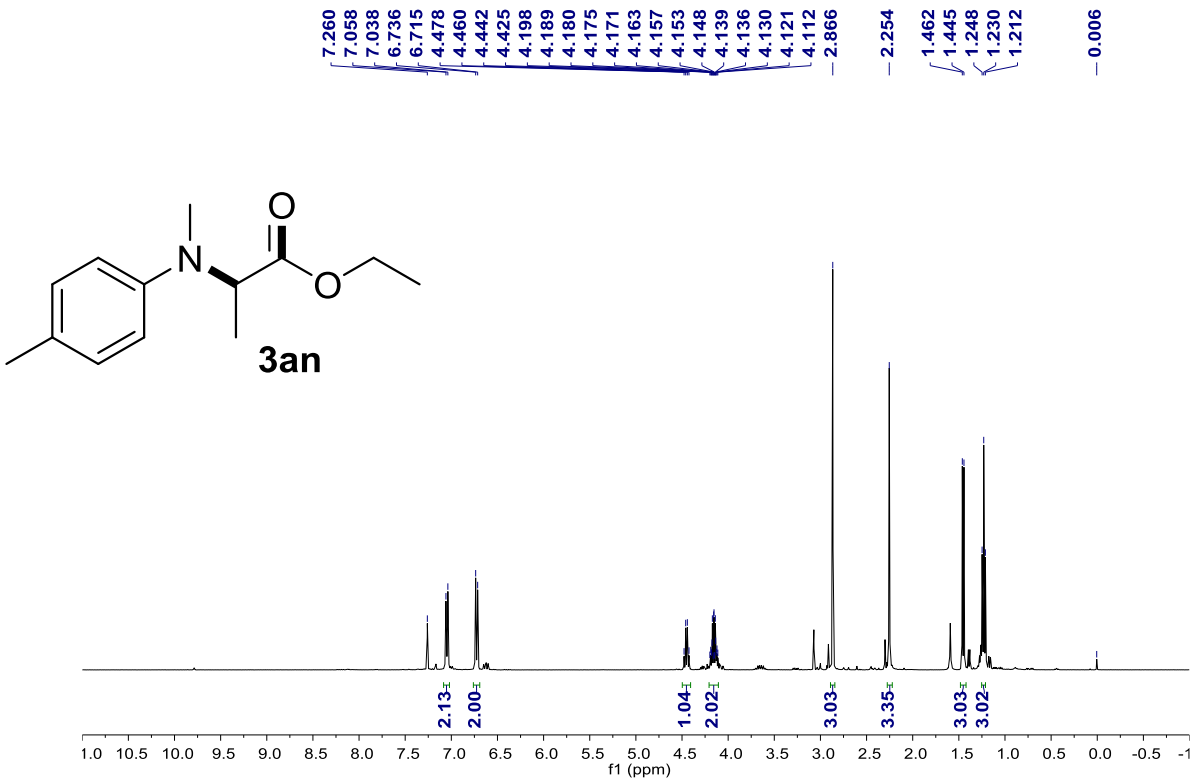
128.08
118.16
114.53
66.26
61.05
42.50
14.16
13.39
3ak
f1 (ppm)

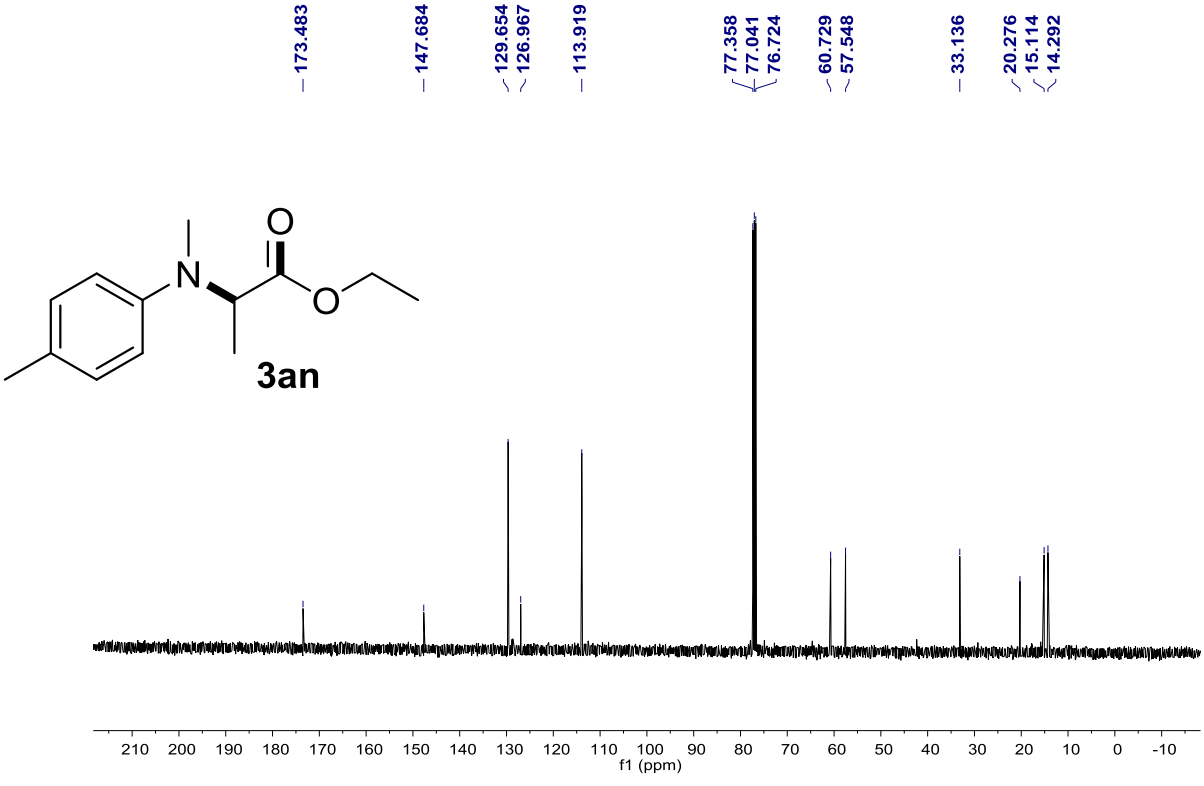

7.260
7.044
7.023
6.700
6.679
4.385
4.367
4.349
4.332
4.192
4.183
4.174
4.165
4.156
4.147
4.138
4.129
4.119
4.112
4.099
4.093
3.382
3.365
3.347
3.329
2.250
2.240
1.485
1.468
1.247
1.229
1.212
1.188
1.170
1.152
0.006
O
N
O
3al
2.09
1.95
1.00
2.04
2.15
3.40
2.76
3.08
3.12
f1 (ppm)

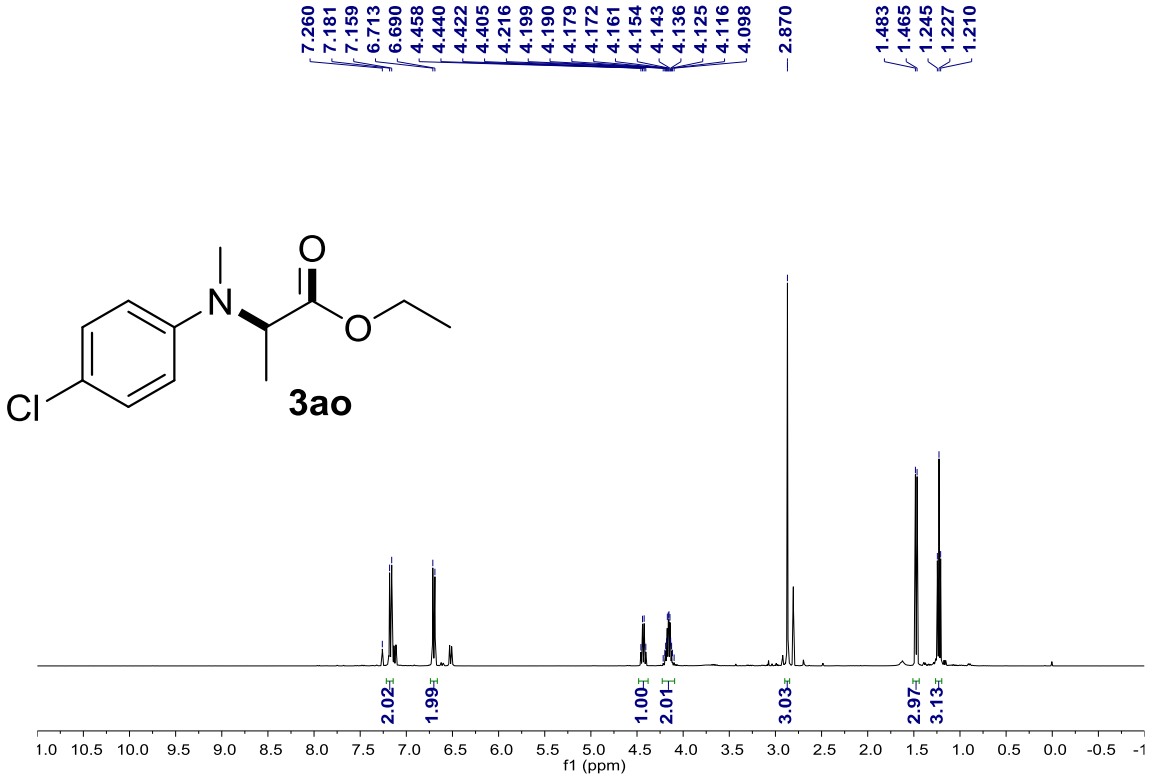

173.965
145.980
129.664
126.787
114.378
77.358
77.040
76.723
60.754
57.646
41.692
20.265
15.935
14.511
14.249
O
N
O
3al
f1 (ppm)

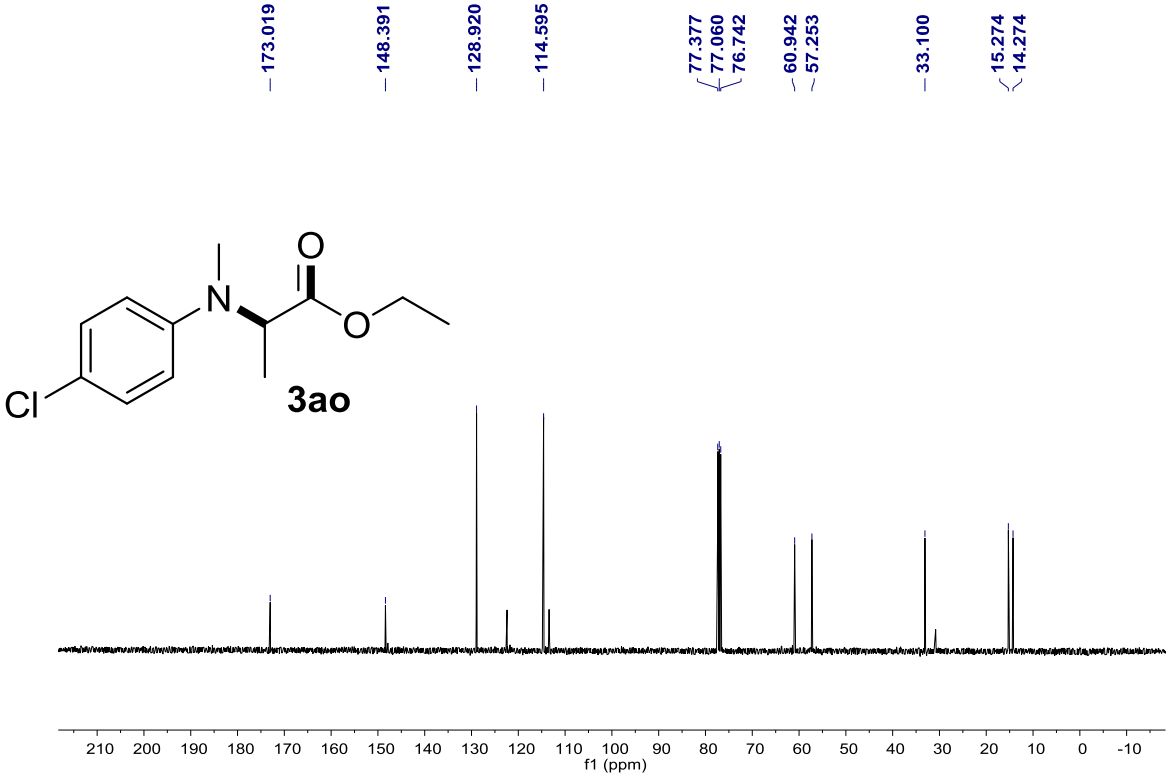

7.260
7.058
7.038
6.736
6.715
4.478
4.460
4.442
4.425
4.198
4.189
4.180
4.175
4.171
4.163
4.157
4.153
4.148
4.139
4.136
4.130
4.121
4.112
2.866
2.254
1.462
1.445
1.248
1.230
1.212
0.006
3am
2.13
2.00
1.04
2.02
3.03
3.35
3.03
3.02
f1 (ppm)

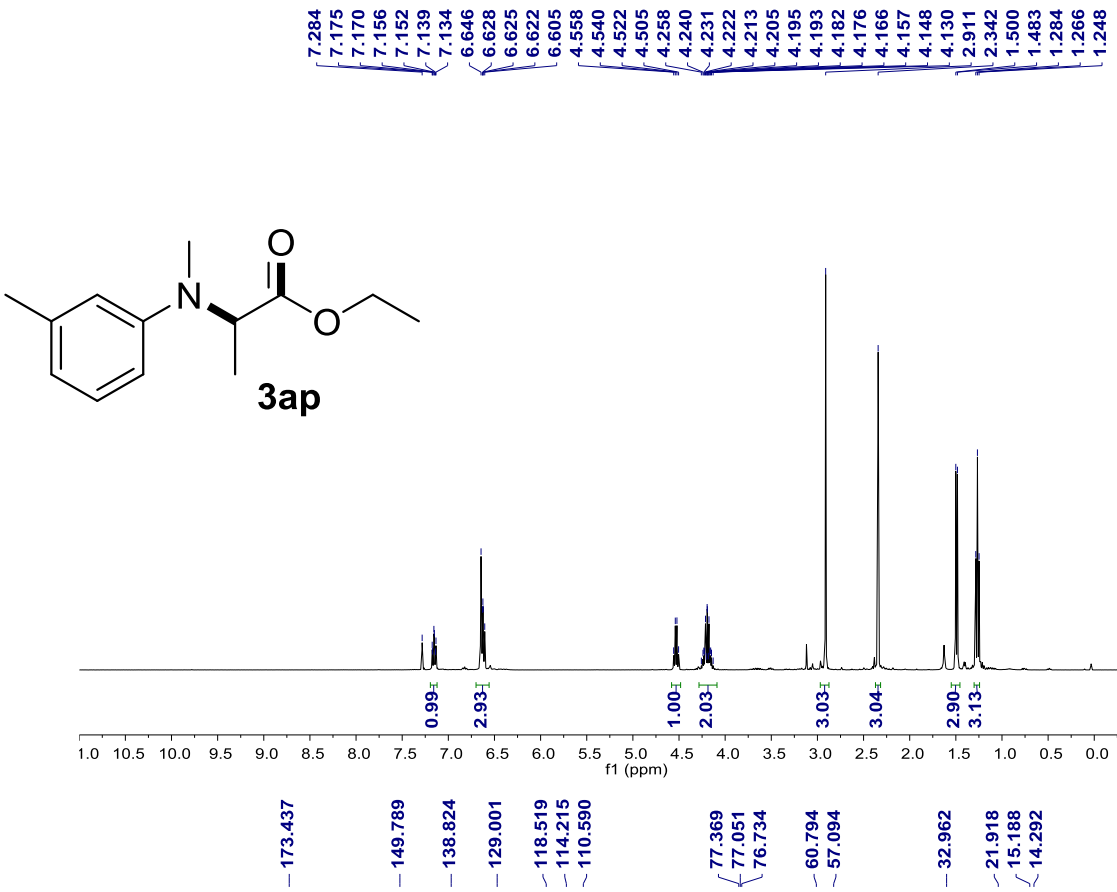

173.26
149.58
129.10
117.60
113.42
60.77
57.08
32.95
15.16
14.22
3am
f1 (ppm)

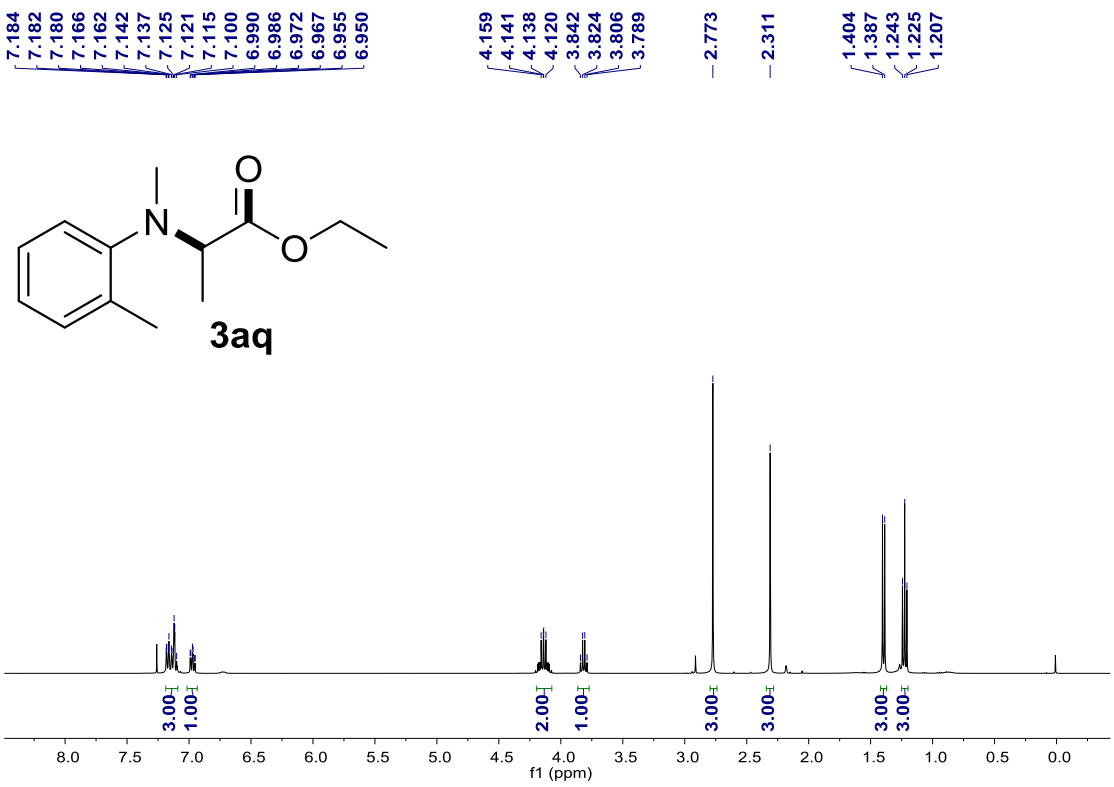

7.260
7.058
7.038
6.736
6.715
4.478
4.460
4.442
4.425
4.198
4.189
4.180
4.175
4.171
4.163
4.157
4.153
4.148
4.139
4.136
4.130
4.121
4.112
2.866
2.254
1.462
1.445
1.248
1.230
1.212
0.006
O
N
O
3an
2.13
2.00
1.04
2.02
3.03
3.35
3.03
3.02
f1 (ppm)

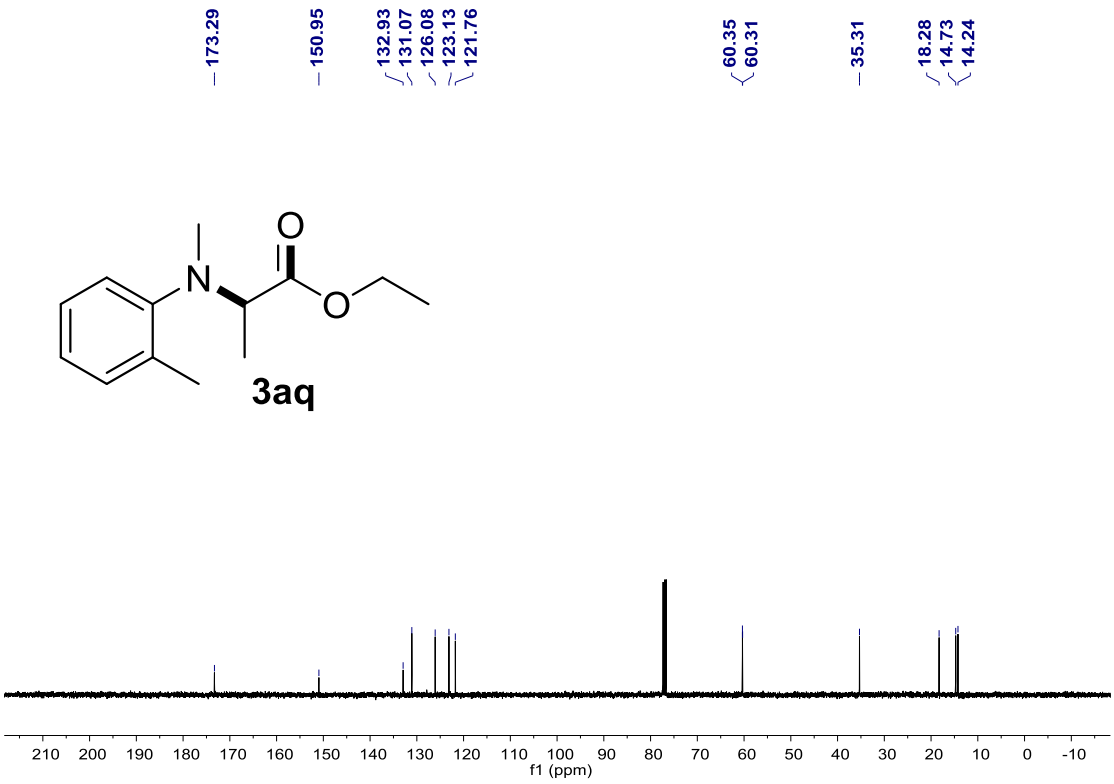

173.483
147.684
129.654
126.967
113.919
77.358
77.041
76.724
60.729
57.548
33.136
20.276
15.114
14.292
O
N
O
3an
f1 (ppm)

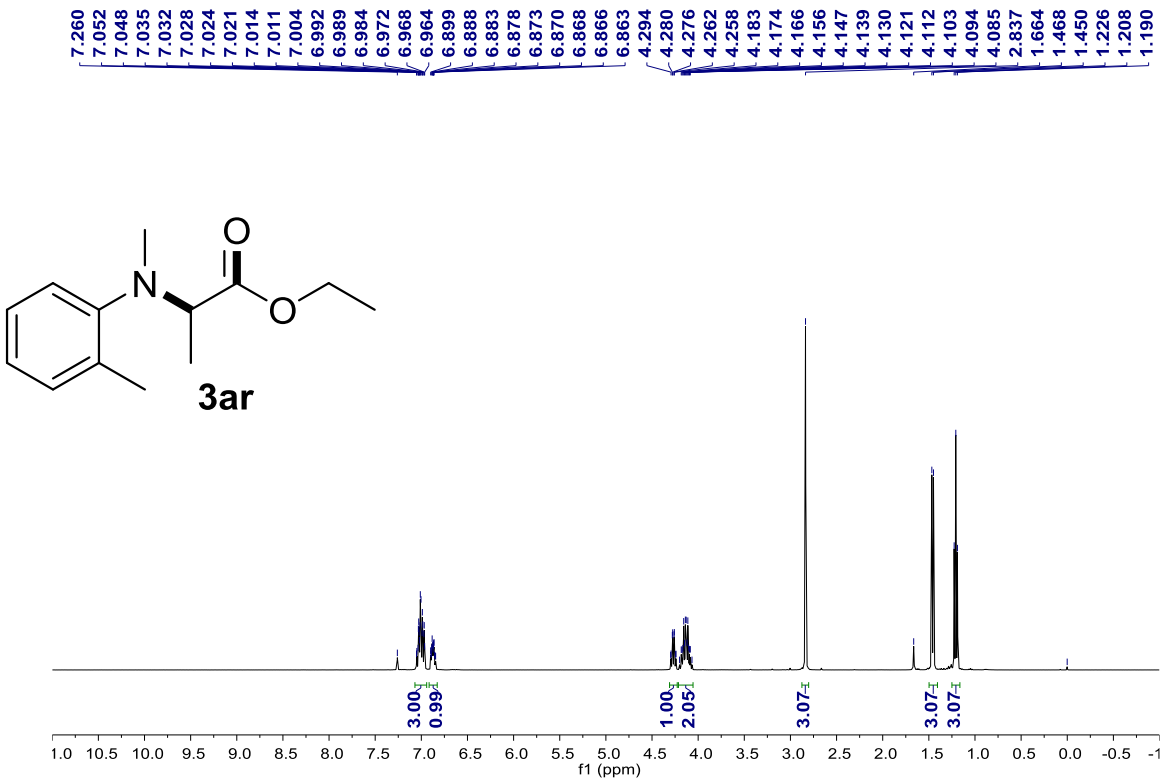

7.260
7.181
7.159
6.713
6.690
4.458
4.440
4.422
4.405
4.216
4.199
4.190
4.179
4.172
4.161
4.154
4.143
4.136
4.125
4.116
4.098
2.870
1.483
1.465
1.245
1.227
1.210
O
N
O
Cl
3ao
2.02
1.99
1.00
2.01
3.03
2.97
3.13
f1 (ppm)

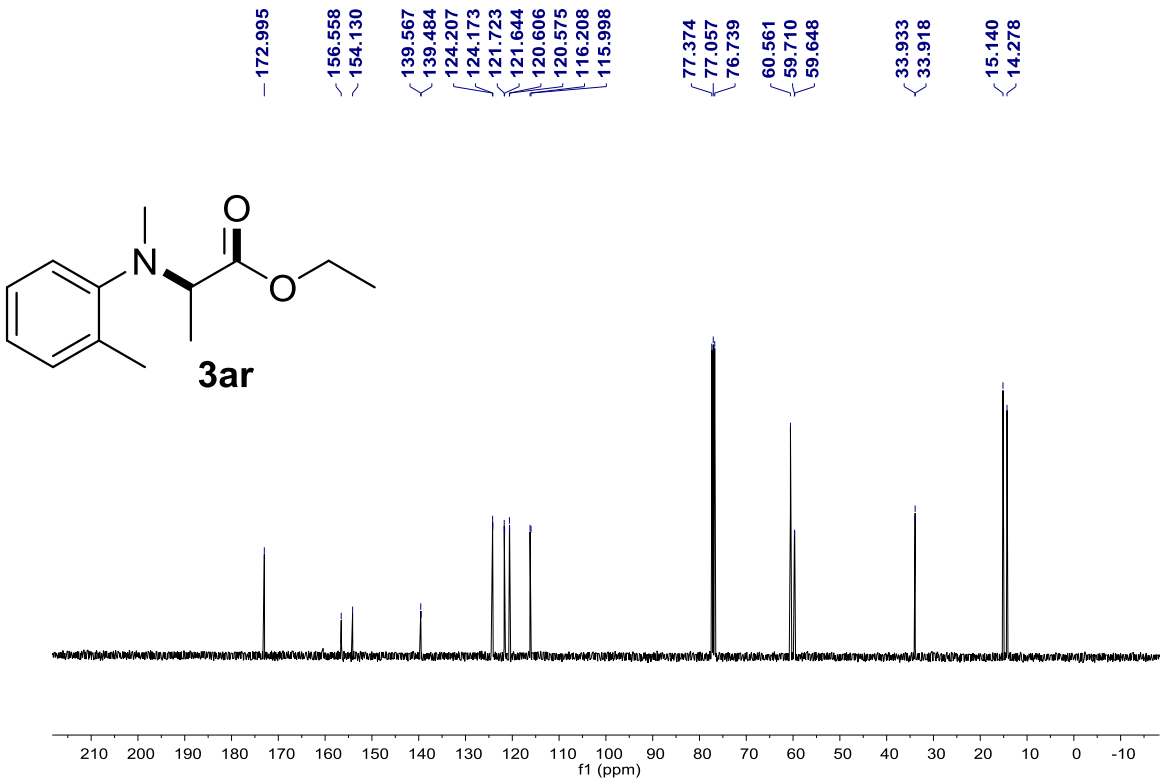

173.019
148.391
128.920
114.595
77.377
77.060
76.742
60.942
57.253
33.100
15.274
14.274
O
N
O
Cl
3ao
f1 (ppm)

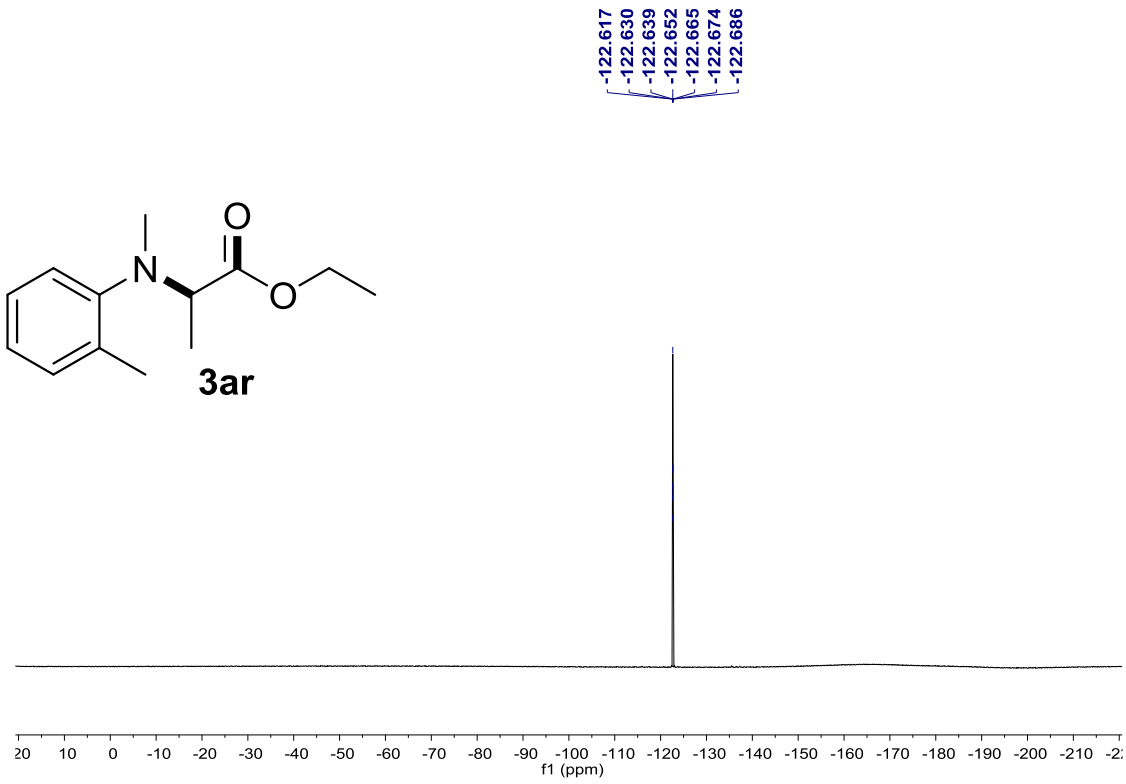

7.284
7.175
7.170
7.156
7.152
7.139
7.134
6.646
6.628
6.625
6.622
6.605
4.558
4.540
4.522
4.505
4.258
4.240
4.231
4.222
4.213
4.205
4.195
4.193
4.182
4.176
4.166
4.157
4.148
4.130
2.911
2.342
1.500
1.483
1.284
1.266
1.248
O
N
O
3ap
0.99
2.93
1.00
2.03
3.03
3.04
2.90
3.13
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
173.437
149.789
138.824
129.001
118.519
114.215
110.590
77.369
77.051
76.734
60.794
57.094
32.962
21.918
15.188
14.292
O
N
O
3ap
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.184
7.182
7.180
7.166
7.162
7.142
7.137
7.125
7.121
7.115
7.100
6.990
6.986
6.972
6.967
6.955
6.950
4.159
4.141
4.138
4.120
3.842
3.824
3.806
3.789
2.773
2.311
1.404
1.387
1.243
1.225
1.207
O
N
O
3aq
3.00
1.00
2.00
1.00
3.00
3.00
3.00
3.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

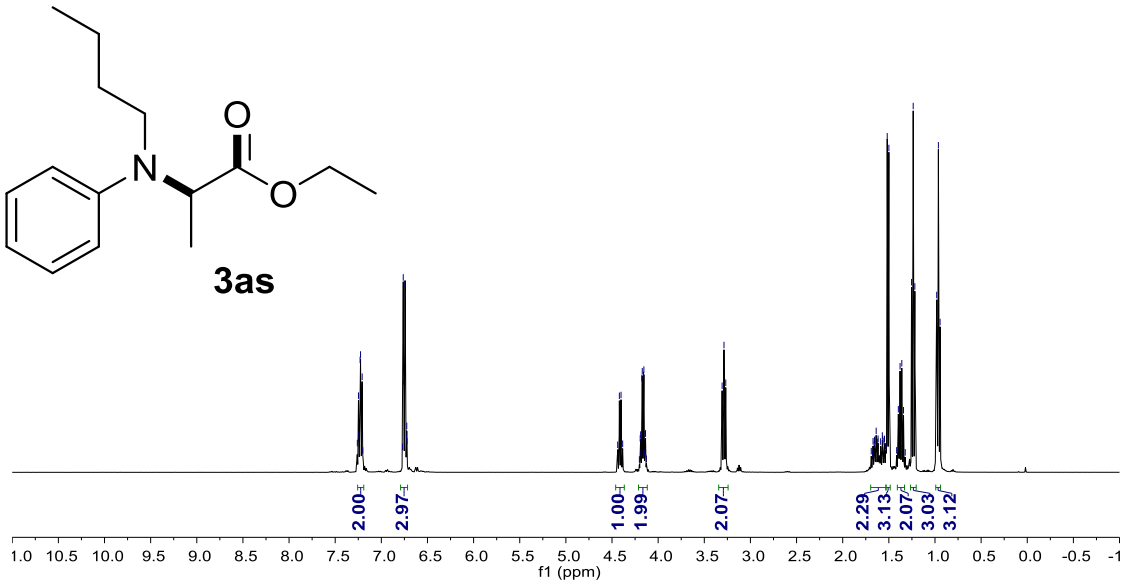

173.29
150.95
132.93
131.07
126.08
123.13
121.76
60.35
60.31
35.31
18.28
14.73
14.24
O
N
O
3aq
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.260
7.052
7.048
7.035
7.032
7.028
7.024
7.021
7.014
7.011
7.004
6.992
6.989
6.984
6.972
6.968
6.964
6.899
6.888
6.883
6.878
6.873
6.870
6.868
6.866
6.863
4.294
4.280
4.276
4.262
4.258
4.183
4.174
4.166
4.156
4.147
4.139
4.130
4.121
4.112
4.103
4.094
4.085
2.837
1.664
1.468
1.450
1.226
1.208
1.190
O
N
O
3ar
3.00
0.99
1.00
2.05
3.07
3.07
3.07
f1 (ppm)

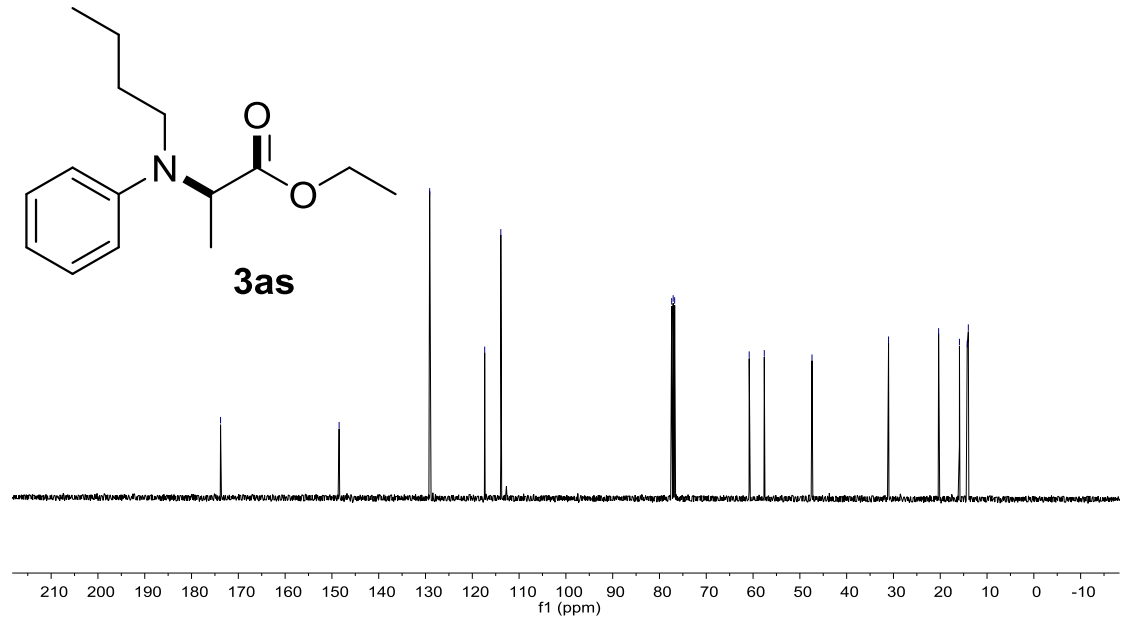

172.995
156.558
154.130
139.567
139.484
124.207
124.173
121.723
121.644
120.606
120.575
116.208
115.998
77.374
77.057
76.739
60.561
59.710
59.648
33.933
33.918
15.140
14.278
O
N
O
3ar
f1 (ppm)

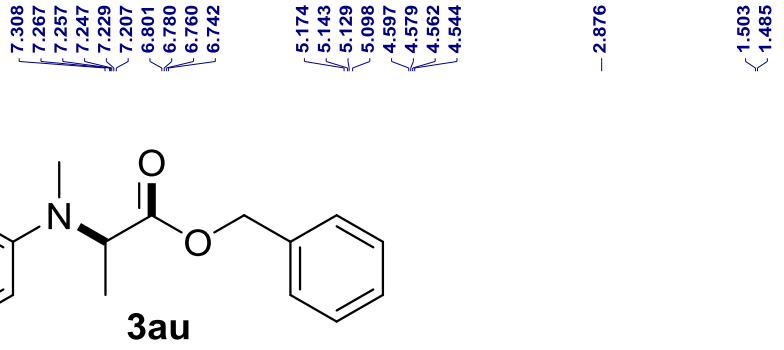

-122.617
-122.630
-122.639
-122.652
-122.665
-122.674
-122.686
O
N
O
3ar
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

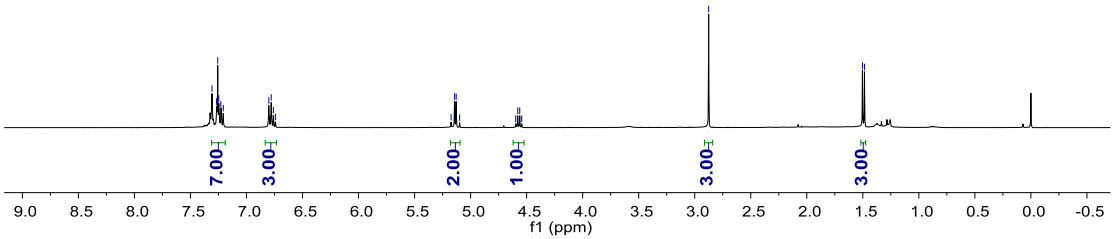
7.248 7.243 7.230 7.225 7.213 7.208 6.763 6.758 6.744 6.741 6.727 6.724 6.722 4.420 4.403 4.194 4.190 4.176 4.172 4.159 4.154 4.141 4.137 3.307 3.291 3.287 3.267 1.671 1.656 1.652 1.637 1.621 1.617 1.589 1.570 1.566 1.553 1.548 1.518 1.500 1.397 1.378 1.360 1.341 1.254 1.236 1.218 0.981 0.963 0.945

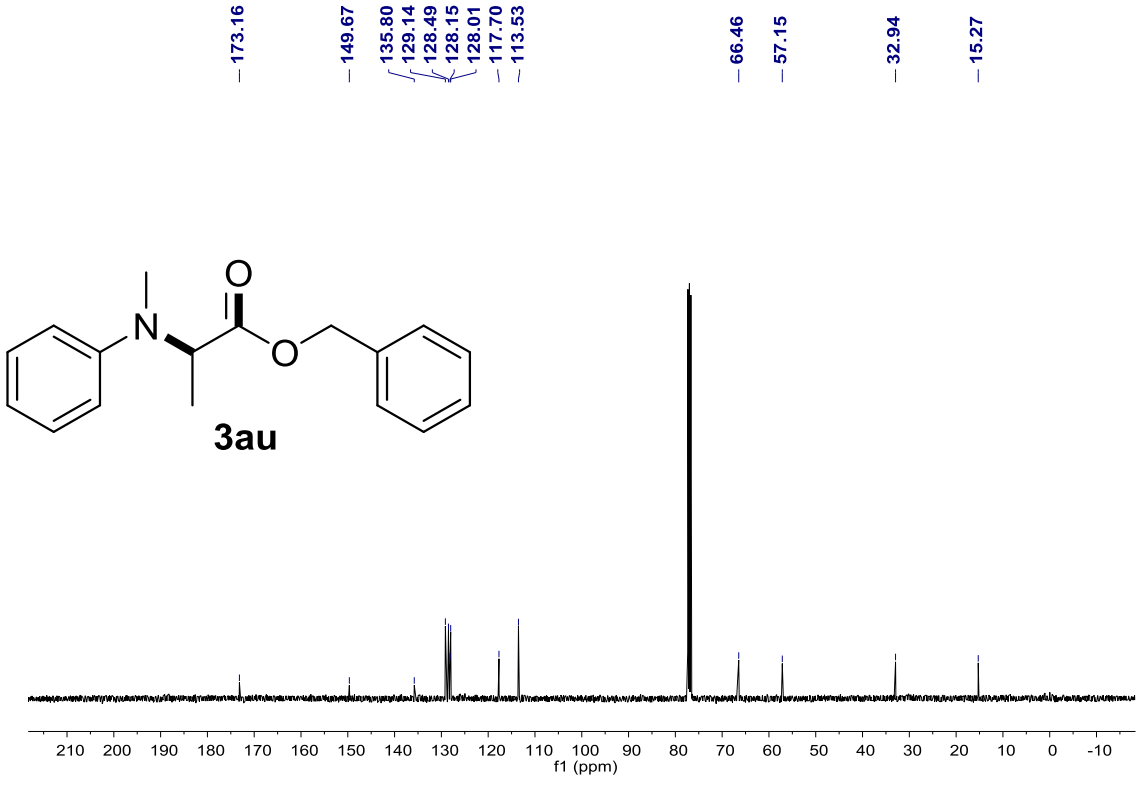


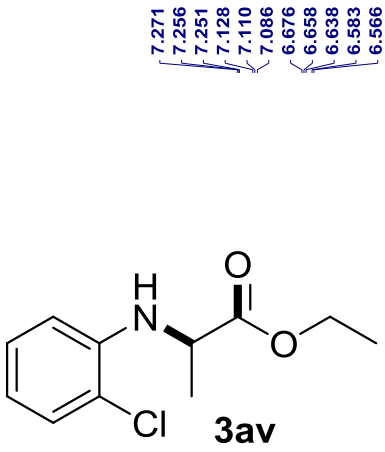
173.796 148.461 129.111 117.350 113.906 77.393 77.074 76.756 60.827 57.602 47.405 31.045 20.348 15.895 14.242 13.999

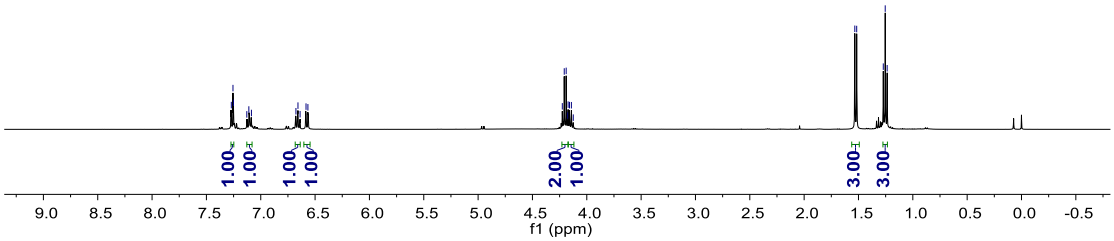


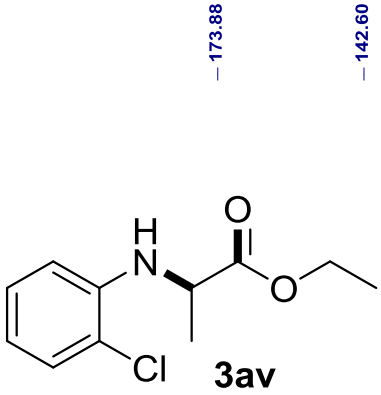

7.308
7.267
7.257
7.247
7.229
7.207
6.801
6.780
6.760
6.742
5.174
5.143
5.129
5.098
4.597
4.579
4.562
4.544
2.876
1.503
1.485
O
N
O
3au

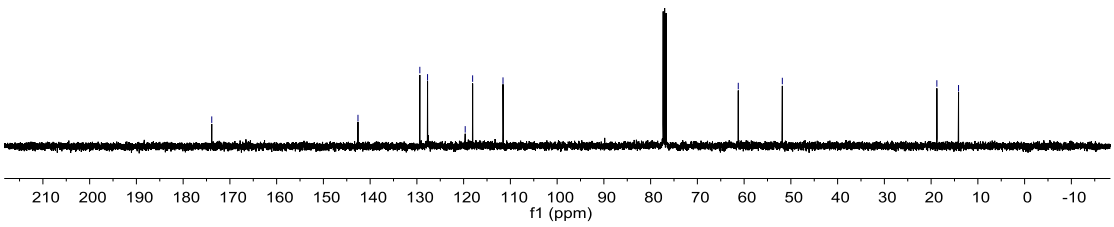

7.00
3.00
2.00
1.00
3.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

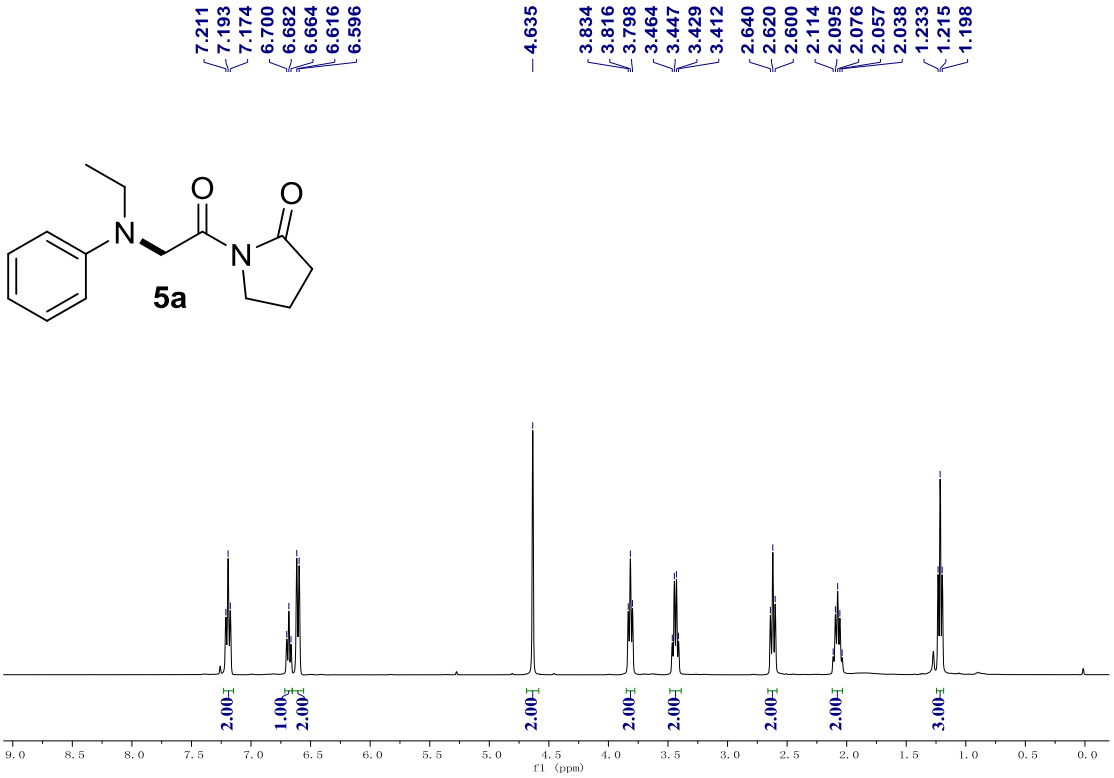

173.16
149.67
135.80
129.14
128.49
128.15
128.01
117.70
113.53
66.46
57.15
32.94
15.27
O
N
O
3au
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.271
7.256
7.251
7.128
7.110
7.086
6.676
6.658
6.638
6.583
6.566
4.225
4.207
4.190
4.176
4.172
4.159
4.142
4.124
1.534
1.516
1.272
1.254
1.236
O
H
N
O
Cl
3av


1.00
1.00
1.00
1.00
2.00
1.00
3.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


173.88
142.60
129.37
127.72
119.67
118.07
111.58
61.25
51.83
18.77
14.13
O
H
N
O
Cl
3av


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.211
7.193
7.174
6.700
6.682
6.664
6.616
6.596
4.635
3.834
3.816
3.798
3.464
3.447
3.429
3.412
2.640
2.620
2.600
2.114
2.095
2.076
2.057
2.038
1.233
1.215
1.198
5a
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1.00
2.00
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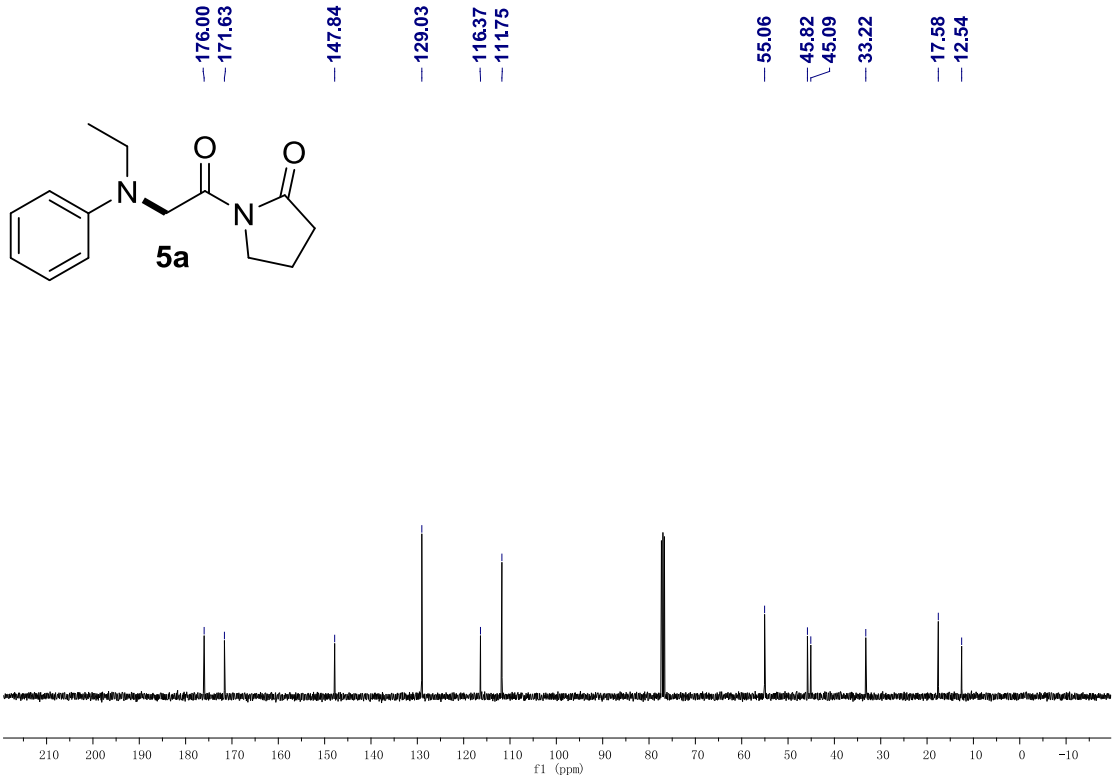
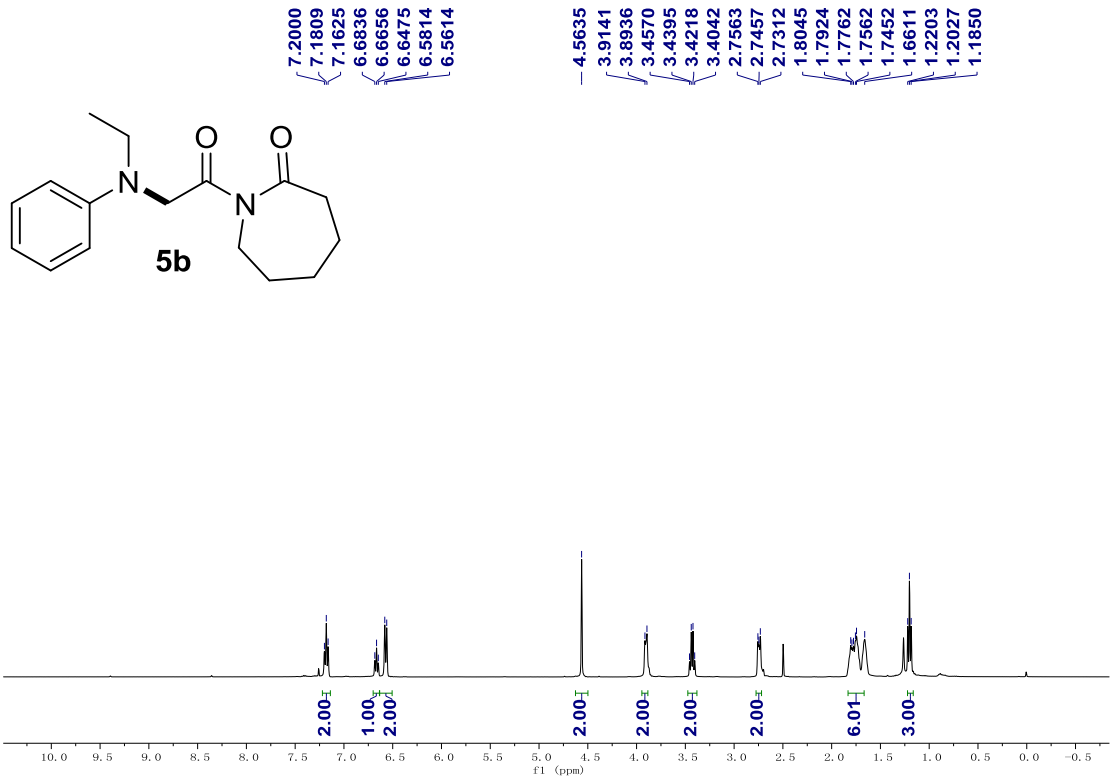
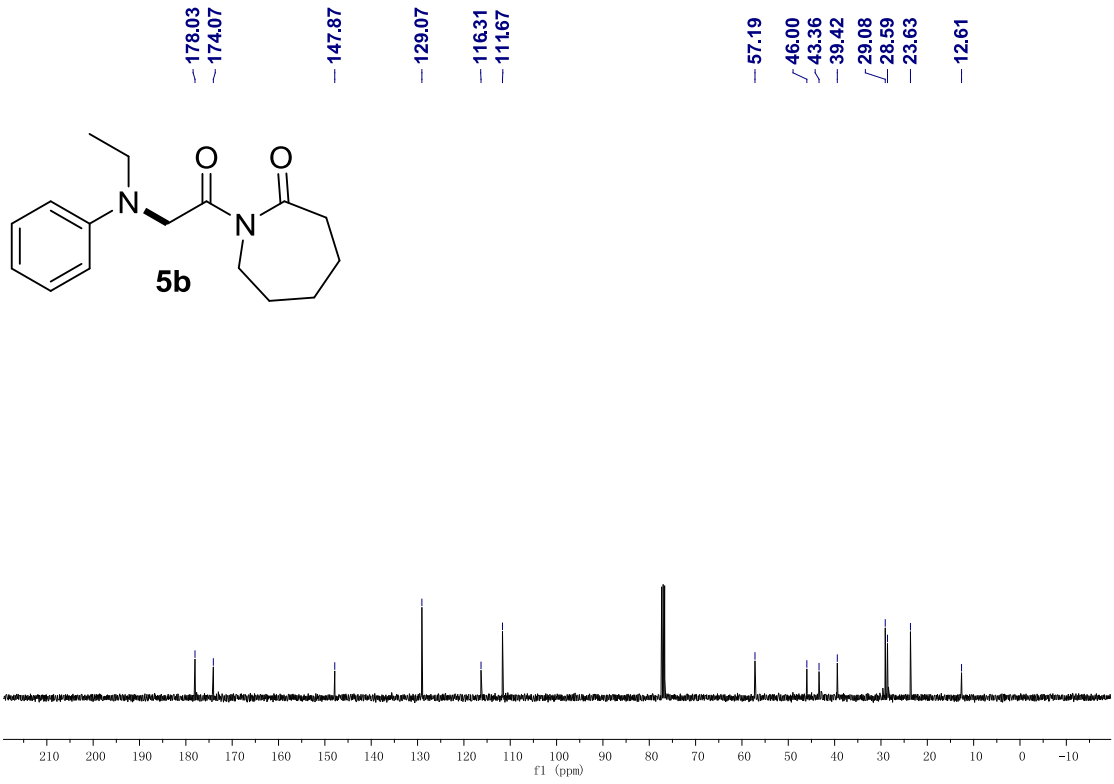
2.00
2.00
2.00
2.00
3.00
f1 (ppm)

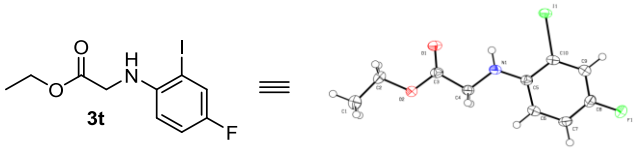

176.00
171.63
147.84
129.03
116.37
111.75
55.06
45.82
45.09
33.22
17.58
12.54
5a
f1 (ppm)


7.2000
7.1809
7.1625
6.6836
6.6656
6.6475
6.5814
6.5614
4.5635
3.9141
3.8936
3.4570
3.4395
3.4218
3.4042
2.7563
2.7457
2.7312
1.8045
1.7924
1.7762
1.7562
1.7452
1.6611
1.2203
1.2027
1.1850
O
O
N
N
5b
2.00
1.00
2.00
2.00
2.00
2.00
2.00
6.01
3.00
f1 (ppm)


178.03
174.07
147.87
129.07
116.31
111.67
57.19
46.00
43.36
39.42
29.08
28.59
23.63
12.61
O
O
N
N
5b
f1 (ppm)

Ⅵ. X-ray Crystallographic Analysis for Product 3t

ORTEP representation with 50 % probability thermal ellipsoids. Crystal data have been deposited to CCDC, number 1962414.



Empirical formula	$C_{10}H_{11}FINO_2$
Formula weight	323.10
Temperature	170 K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 7.5850(5) Å alpha = 100.883(2) deg. b = 7.9584(4) Å beta = 93.120(2) deg. c = 10.8239(6) Å gamma = 118.456(2) deg.
Volume	556.16(6) $Å^3$
Z, Calculated density	2, 1.929 Mg/m^3
Absorption coefficient	2.873 mm^{-1}
F(000)	312.0
Crystal size	0.19 × 0.12 × 0.08 mm
Theta range for data collection	6.006 to 52.904 deg.
Limiting indices	$-9 \leq h \leq 9$, $-9 \leq k \leq 9$, $-12 \leq l \leq 13$
Reflections collected / unique	6262 / 2248 [Rint = 0.0250, Rsigma =0.0274]
Completeness to theta = 25.242	99.9%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2248 / 0 / 137
Goodness-of-fit on F^2	1.069
Final R indices [I>=2σ (I)]	R1 = 0.0180, wR2 = 0.0401
R indices (all data)	R1 = 0.0198, wR2 = 0.0410